





Y16-11-10-10

Jan

Chemistry of selenophene. IV. Iodination and metal-  
lation of selenophene and its homologs. Selenophene car-  
boxylic acids. Yu. K. Ivanov and N. K. Sadovaya (State  
university, Kazan, USSR) *Chem. Abstr.* 20, 3154-7 (1956)  
Selenophene (1.6 g) in 25 ml.  $\text{C}_6\text{H}_6$   
was heated with 1.0 g. iodine and a 7  
g. solution of  $\text{Na}_2\text{S}_2\text{O}_3$  in 10 ml.  $\text{C}_6\text{H}_6$  and 10 ml.  
water, and the mixture was stirred over  $\text{HgO}$  to give  
a black solid, which was washed with water, dried, and  
recrystallized from  $\text{C}_6\text{H}_6$ . mp. 84-86°.  $\text{C}_{10}\text{H}_6\text{S}_2\text{O}_2$   
calcd. for  $\text{C}_{10}\text{H}_6\text{S}_2\text{O}_2$ : C, 54.5%; H, 2.7%; S, 11.9%.  
Found: C, 54.5%; H, 2.7%; S, 11.9%. The solid was  
recrystallized from  $\text{C}_6\text{H}_6$  and the mother liquor was  
evaporated. Total yield 0.5 g. mp. 84-86°.  $\text{C}_{10}\text{H}_6\text{S}_2\text{O}_2$   
calcd. for  $\text{C}_{10}\text{H}_6\text{S}_2\text{O}_2$ : C, 54.5%; H, 2.7%; S, 11.9%.  
Found: C, 54.5%; H, 2.7%; S, 11.9%. The solid was  
recrystallized from  $\text{C}_6\text{H}_6$  and the mother liquor was  
evaporated. Total yield 0.5 g. mp. 84-86°.  $\text{C}_{10}\text{H}_6\text{S}_2\text{O}_2$   
calcd. for  $\text{C}_{10}\text{H}_6\text{S}_2\text{O}_2$ : C, 54.5%; H, 2.7%; S, 11.9%.  
Found: C, 54.5%; H, 2.7%; S, 11.9%.

[illegible]





YUR'YEV, Yu.K.

Mutual catalytic transformations of heterocyclic compounds. New methods  
for the synthesis of heterocyclic compounds. Uch.zap.Mosk. un.  
no.175:159-200 '56. (MIRA 10:3)\*  
(Heterocyclic compounds)

PHASE I BOOK EXPLOITATION 779

Yur'yev, Yuriy Konstantinovich

Prakticheskiye raboty po organicheskoy khimii. [vyp. 1]: Sintezy s pomoshch'yu tsink-i magniyorganicheskikh soedineniy, polimerizatsiya i depolimerizatsiya polikondensatsiya (Laboratory Work in Organic Chemistry. Nr. 1: Syntheses With Organic Zinc and Magnesium Compounds; Polymerization and Depolymerization; Polycondensation.) Moscow, Izd-vo Moskovskogo Univ-ta, 1957. 126 p. 12,000 copies printed.

Ed.: Korobitsyna, I.K.; Tech. Ed.: Lomilina, L.N.

PURPOSE: This book is a handbook for laboratory work in organic chemistry intended for university students specializing in chemistry.

COVERAGE: This book is the first issue of the series "Laboratory Work in Organic Chemistry", based on the laboratory course in organic chemistry given at Moscow State University in conjunction with the lecture course "Synthetic Methods of Organic Chemistry". The laboratory course is considered an independent course rather than a

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supplement to the lecture course. The programs of both the laboratory and lecture courses were worked out by the faculty members of the Chair of Organic Chemistry, headed previously by Academician N.D.Zelinskiy and at present by Academician A.N.Nesmeyanov. The first issue consists of two parts: "Syntheses With Organic Zinc and Magnesium Compounds" by Yu.K.Yur'yev and R.Ya.Levina; and "polymerization and Depolymerization Condensation" by Yu.K.Yur'yev. There are no references.

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SYNTHESES WITH ORGANIC ZINC AND MAGNESIUM COMPOUNDS by Yu.K.Yur'yev and R.Ya.Levina	
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Obtaining magnesium organic compounds	9
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YUR'YEV, Yu.K.; MEZENTSOVA, N.N.; SADOVAYA, N.K.

Progress in the chemistry of selenophene. Vest. Mosk.un.Ser.mat.  
mekh. astron. fiz. khim. 12 no.4:201-222 '57. (MIRA 11:5)

1.Kafedra organicheskoy khimii Moskovskogo gosudarstvennogo  
universiteta.

(Selenophene)

**AUTHORS:**

Yuryev, Yu. K., and Yelyakov, G. B.

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**TITLE:**

Tetraacyloxysilanes in Organic Synthesis. Part 10. Silicon Anhydrides of Monobasic Saturated-Organic Acids in the Synthesis of Alkyl-Beta-Vinyl Chloride Ketones (Tetraatsiloksisilany v organicheskom sinteze. X. Kremneangidridy odnoosnovnykh predel'nykh-organicheskikh kislot v sinteze alkil-beta-khlorvinilketonov)

**PERIODICAL:**

Zhurnal Obshchey Khimii, 1957, Vol. 27, No. 1, pp. 176-179 (U.S.S.R.)

**ABSTRACT:**

Into the reaction of acetylene taking place in the presence of anhydrous aluminum chloride, the authors introduced silicon anhydrides of acetic, propionic, n-butyric and n-caproic acids and obtained homologous alkyl-beta-vinyl chloride ketones amounting to 30-41% of the amount of acid submitted to reaction. The yields were lower than the ones obtainable with acid chlorides. If the acetylene would have reacted not only with the silicon anhydride but also with the acid chloride which could have been formed from the former under effect of the aluminum chloride then the introduction of hydrogen chloride into the reaction mixture would have brought a greater yield of alkyl-beta-vinyl chloride ketone. The reaction of tetraacyloxysilanes with acetylene in

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## Tetraacyloxysilanes in Organic Synthesis

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the presence of  $\text{AlCl}_3$  was established to be rather an addition reaction. The addition of the silicon anhydride saturated monobasic organic acid to the acetylene led to the formation of orthosilicic acid ester and alkyl-beta-oxyvinyl ketone which under the effect of  $\text{AlCl}_3$  submitted to cleavage with the resulting formation of methyl-beta-vinyl chloride ketone and aluminum halide salt of orthosilicic acid. It was assumed that silicon anhydrides of saturated monobasic organic acids react with acetylene in the presence of anhydrous aluminum chloride in accordance with the Kondakov type reaction (7), i.e. directly, and that the formation of alkyl-beta-vinyl chloride ketones is due to the cleavage by  $\text{AlCl}_3$  of the initial addition products - orthosilicic acid esters and alkyloxyvinyl ketones.

There are 10 references, of which 9 are Slavic.

ASSOCIATION: The Moscow State University (Moskovskiy Gosudarstvennyy Universitet)

PRESENTED BY:

SUBMITTED: February 17, 1956

AVAILABLE:

Card 2/2

474

**AUTHORS:** Yuryev, Yu. K., and Mezentsova, N. N.

**TITLE:** The Chemistry of Selenophene. Part 5. Selenophene-2-Aldehyde, Selenophene-2-Carbinol and Selenophene-2-Acrylic Acid (Khimiya Selenofena. V. Selenofen-2-aldegid, selenofen-2-karbinol i selenofen-2-akrilovaya kislota)

**PERIODICAL:** Zhurnal Obshchey Khimii, 1957, Vol. 27, No. 1, pp. 179-182 (U.S.S.R.)

**ABSTRACT:** Using dimethylformamide as a base, the authors synthesized a hitherto unknown selenophene-2-aldehyde which is similar in its properties to aromatic aldehyde. The formylation of selenophene with dimethylformamide was smooth, giving a 75% aldehyde yield. Oxidation of selenophene-2-aldehyde with hydrogen peroxide resulted in the formation of selenophene-2-carboxylic acid; heating of selenophene-2-aldehyde with acetic anhydride and anhydrous sodium acetate gave selenophene-2-acrylic acid. This acid was also obtained through condensation of selenophene-2-aldehyde with malonic acid in the presence of pyridine with consequent decarboxylizing of the forming alpha-carboxy-beta-(2-selenophene)-acrylic acid. Selenophene-2-aldehyde (when reduced with formaldehyde in conditions of the Tishchenko-Cannizzaro reaction)

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1957-1960

Ethylene anhydride in the synthesis of 2-phenyl-2-oxirane compounds with two olefin atoms. The reaction of 2-alkyl-1,4-bis(phenyl)phenylene and 2-phenyl-1,4-bis(phenyl)phenylene with ethylene anhydride at 180-185°C. gave 2-phenyl-2-oxirane in 10-20% yield. At 180-185°C. in the presence of 50% aqueous sodium hydroxide solution, 2-phenyl-2-oxirane was obtained in 10-20% yield. The reaction of 2-phenyl-1,4-bis(phenyl)phenylene with ethylene anhydride in the presence of 50% aqueous sodium hydroxide solution at 180-185°C. gave 2-phenyl-2-oxirane in 10-20% yield. The reaction of 2-phenyl-1,4-bis(phenyl)phenylene with ethylene anhydride in the presence of 50% aqueous sodium hydroxide solution at 180-185°C. gave 2-phenyl-2-oxirane in 10-20% yield.

1  
2 May  
3



YURINOV 10/15

Director: 4843/4131

1. On 10/15/86, the Director of the CIA, Mr. William J. Casey, received a letter from the Soviet Ambassador to the United States, Mr. Anatoly Dobrynin, regarding the activities of the Soviet intelligence service in the United States.

2. The letter stated that the Soviet intelligence service had been conducting extensive activities in the United States for many years, and that it had been successful in obtaining a large amount of information about the United States government and its policies.

3. The letter also stated that the Soviet intelligence service had been conducting extensive activities in the United States for many years, and that it had been successful in obtaining a large amount of information about the United States government and its policies.

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8. The letter stated that the Soviet intelligence service had been conducting extensive activities in the United States for many years, and that it had been successful in obtaining a large amount of information about the United States government and its policies.

9. The letter also stated that the Soviet intelligence service had been conducting extensive activities in the United States for many years, and that it had been successful in obtaining a large amount of information about the United States government and its policies.

10. The letter stated that the Soviet intelligence service had been conducting extensive activities in the United States for many years, and that it had been successful in obtaining a large amount of information about the United States government and its policies.

YUR'YEV, Yu.K.

YUR'YEV, Yu.K.; MEZENTSOVA, N.M.; MELENT'YEVA, T.A. TRESHCHOVA, Ye.G.

The chemistry of selenophene. Part 7: Synthesis and acetylation  
of 3-arylselenophenes and 2,3-benzoselenophene. Zhur. ob. khim.  
27 no.8:2260-2267 Ag '57. (MLRA 10:9)

1. Moskovskiy gosudarstvennyy universitet.  
(Selenophene)

YUR'YEV, Yu.K.; MEZENTSOVA, N.N.; BALASHOVA, T.A.

Chemistry of selenophene. Part 8: N-(selenenal-2) - amines,  
2-phenyl-4-(selenenal-2)- oxazolone-5, 5-(selenenal-2 - thiazolidone  
-4- thion-2 selenenal-2-rodanine) and hydrazothiazolynon selenophene-  
2-aldehyde. zhur. ob. khim. 27 no.9:2536-2541 S '57. (MIRA 11:3)

1. Moskovskiy gosudarstvennyy universitet.  
(Selephone)

YUR'YEV, Yu.K.; DYATLOVITSEAYA, S.V.

Ethylenesulfide in the synthesis of heterocyclic compounds with two heteroatoms. Part 3: -arylthiazolidones-2 from N-B-mercapto-ethyl) -arylamines. Zhur.ob.khim. 27 no.10:2644-2648 0 '57.  
(MIRA 11:4)

1.Moskovskiy gosudarstvennyy universitet.  
(Thiazoldinone) (Amines)

YUR'YEV, Yu. K.

79-11-49/56

AUTHORS: Yur'yev, Yu. K., Dyatlovitskaya, S. V.  
 TITLE: Ethylene Sulfide in the Synthesis of Heterocyclic Compounds With two Heteroatoms (Etilensulfid v sinteze Geterotsiklicheskikh soedineniy s dvumya Geteroatomami).  
 IV. Synthesis of 3-Aryl-Thiazolidines and 4-Aryl-thiazanes-1,4 (IV. Sintez 3-aryl-tiazolidinov i 4-ariltiazanov-1,4).

PERIODICAL: Zhurnal Obshchey Khimii, 1957, Vol. 27, Nr 11, pp. 3148-3151 (USSR)

ABSTRACT: In the preceding paper the authors showed that N-( $\beta$ -mercaptoethyl)-arylamines readily enter into reaction with the chloranhydride of carbonic acid by forming 3-arylthiazolidones-2 with yields of 50-90%. In connection with this it was of interest to investigate the behavior of the N-( $\beta$ -mercaptoethyl)-arylamines in an analogous reaction with dihalides, with less movable halogen atoms than in phosgene, i.e. with 1,1- and 1,2-dialkyl-halides. In publications it is pointed out that bromethylene was already used in the condensation with  $\beta$ -aminoethylmercaptan, but it seems that thiazane-1,4 on that occasion forms only in a small amount. The condensation of N-( $\beta$ -mercaptoethyl)-aniline, N-( $\beta$ -mercapto-

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Ethylene Sulfide in the Synthesis of Heterocyclic Compounds 79-11-49/56  
With two Heteroatoms. IV. Synthesis of 3-Aryl-Thiazolidines and  
4-Arylthiazanes-1,4

ethyl)-p-toluidine, N-(β-mercaptoethyl)-o-toluidine,  
N-(β-mercaptoethyl)-m-anisidine and N-(β-mercaptoethyl)-  
o-anisidine with bromomethylene leads to the formation of a  
thiazolidine-cycle where, correspondingly, 3-phenyl-,  
3-m-tolyl-, 3-m-anisyl- and 3-o-tolyl- as well as the  
hitherto unknown 3-o-anisyl-thiazolidines form. The same  
reaction of N-(β-mercaptoethyl)-aniline, N-(β-mercaptoethyl)-  
m-toluidine, N-(β-mercaptoethyl)-o-anisidine and N-(β-  
mercaptoethyl)-m-anisidine with bromethylene causes the  
formation of a thiazane-cycle -1,4 on which occasion the  
4-phenyl-, 4-m-tolyl-, 4-m-anisyl- and 4-o-anisylthiazanes  
are obtained.

There are 7 references, 3 of which are Slavic.

ASSOCIATION: Moscow State University (Moskovskiy gosudarstvennyy universitet).

SUBMITTED: November 5, 1956

AVAILABLE: Library of Congress

Card 2/2

1. Cyclic compounds-Synthesis
2. Ethylene sulfide-Chemical reactions
3. Cyclic compounds-Condensation reactions

YUR'YEV, Yu. K.

79-11-50/56

AUTHORS:

Yur'yev, Yu. K., Dyatlovitskaya, S. V.

TITLE:

Ethylene Sulfide in the Synthesis of Heterocyclic Compounds  
With two Hetero-Atoms (Etilensul'fid v sinteze geterotsikli-  
cheskikh soyedineniy s dvumya geteroatomami).

PERIODICAL:

Zhurnal Obshchey Khimii, 1957, Vol. 27, Nr 11,  
pp. 3152-3154 (USSR)

ABSTRACT:

The authors previously showed that N-( $\beta$ -mercaptoethyl)-  
arylamines readily enter into condensation with aldehydes,  
phosgene, and bromethylene and correspondingly form 2-alkyl-  
(or aryl)-3-arylthiazolidines, 3-arylthiazolidines-2,  
3-arylthiazolidines and 4-arylthiazanes-1,4. But N-( $\beta$ -  
mercaptoethyl)-arylamines cannot only be used in the above-  
mentioned condensation, i.e. with 2 splittings off of water,  
hydrogen chloride and hydrogen bromide, but also in a  
condensation with splitting off of hydrogen sulfide, e.g.  
in the reaction with carbon disulfide which should  
necessarily lead to the formation of 3-arylthiazolidinthione-2.  
Of the compounds of the thiazolidinthion-2 group those having  
no substituents on the nitrogen-atom are best investigated,  
the 3-alkyl-(or aryl)-thiazolidinthions-2 worst. In the

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Ethylene Sulfide in the Synthesis of Heterocyclic  
Compounds With two Hetero-Atoms

79-11-50/56

present paper the condensation of N-( $\beta$ -mercaptoethyl)-arylamines with carbon disulfide was carried out. This condensation led to 3-arylthiazolidinthionene-2, which convincingly indicates the high reactivity of  $\beta$ -amino-mercaptan, arylated on nitrogen, introduced by the authors into the reaction (see formula). By conversion of 3-arylthiazolidinthions-2 to the corresponding 3-arylthiazolidones-2 by mercuric oxide their structure was proved. There are 13 references, 5 of which are Slavic.

ASSOCIATION: Moscow State University (Moskovskiy gosudarstvennyy universitet).

SUBMITTED: November 12, 1956

AVAILABLE: Library of Congress

Card 2/2

1. Cyclic compounds-Synthesis
2. Ethylene sulfide-Chemical reactions
3. N-( $\beta$ -mercaptoethyl)-arylamines-Condensation reactions
4. Carbon disulfide-Condensation reactions



Yur'yev, Yu. K.

79-11-51/56

AUTHORS: Yur'yev, Yu. K., Mezentsova, N. N.,  
Vas'kovskiy, V. Ye.

TITLE: Chemistry of Selenophene (Khimiya selenofena).  
IX. Condensation of Selenophene-2-Aldehyde With Methylketones.  
Synthesis and Reactions of 2-Methylselenophene-5-Aldehyde  
(IX. Kondensatsiya selenofen-2-al'degida s metilketonami.  
Sintez i reaktsii 2-metilselenofen-5-al'degida).

PERIODICAL: Zhurnal Obshchey Khimii, 1957, Vol. 27, Nr 11,  
pp. 3155-3160 (USSR)

ABSTRACT: In the present paper the authors continue the investigation  
of the reactivity of selenophene-2-aldehyde in examples of  
its condensation with methylketones. Its condensations with  
methylketones proceed smoothly and lead to the formation  
of unsaturated ketones which possess the selenophene-cycle.  
In this manner the following compounds were obtained:  
selenenal-2-acetone,  $\alpha$ -(selenenal-2)-acetophenone,  
 $\alpha$ -(selenenal-2)- $\gamma$ -methylacetophenone, 1-phenyl-5-  
(selenienyl-2)-pentadiene-1,4-on-3, 1-(furyl-2)-5-  
(selenienyl-2)-pentadiene-1,4-on-3 and 1,5-di-(selenienyl-2)-  
pentadiene-1,4-on-3. The aminomethylation of selenenal-2-  
acetone according to Mannich (Mannikh) leads to the hydro-

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Chemistry of Selenophene. IX. Condensation of Selenophene-2- Aldehyde With Methylketones. Synthesis and Reactions of 2-Methylselenophene-5-Aldehyde 79-11-51/56

chloride of 5-dimethylamino-1-(selenienyl-2)-pentene-1-ols-3. The reduction of selenophene-2-aldehyde and 2-methylselenophene-5-aldehyde according to Kizhner leads to 2-methylselenophene and correspondingly to 2,5-dimethylselenophene. The condensation of 2-methylselenophene-5-aldehyde with hippuric acid, rhodanine and malonic acid correspondingly yields 2-phenyl-4-(2-methylselenenal-5)-oxazolone-5,5-(2-methylselenenal-5)-thiazolidone-4-thion-2 and  $\beta$ -(2-methylselenophene-5)-acrylic acid. The condensation of thiosemicarbazone of 2-methylselenophene-5-aldehyde with chloroacetic acid leads to the hydrazothiazolinone of 2-methylselenophene-5-aldehyde. There are 4 references, all of which are Slavic.

ASSOCIATION: Moscow State University (Moskovskiy gosudarstvennyy universitet).

SUBMITTED: November 14, 1956

1. Selenophene-2-aldehyde-Condensation reactions
2. Methylketones-Condensation reactions
3. 2-Methylselenophene-5-aldehyde-Synthesis
4. 2-Methylselenophene-5-aldehyde-Condensation reactions

Card 2/2

AUTHORS: Yur'yev, Yu. K., Belyakova, Z. V., Zefirov, N. S. 79-12-19/43

TITLE: Tetraacyloxysilanes in Organic Synthesis  
(Tetraatsiloksisilany v organicheskom sinteze).  
X. Comparative Effect of Catalysts on the Occasion of Acylation  
Reaction of Benzene and Thiophene With Tetraacyloxysilanes  
(Sravnitel'noye deystviye katalizatorov v reaktsii atsiliro-  
vaniya benzola i tiofena tetraatsiloksisilanami).

PERIODICAL: Zhurnal Obshchey Khimii 1957, Vol. 27, Nr 12, pp. 3264-3270  
(USSR)

ABSTRACT: In the present work the comparative effect of a series of  
catalysts in the acylation reaction of thiophene with tetra-  
acetoxyasilane with the mixed anhydride of the orthosilicic  
acid and acetic acid is investigated. The cabality of reacting  
of the two anhydrides to be expected was examined in order  
to known whether the actual acylation of thiophene, selenophene  
and benzene is due to the silico-anhydrides of the organic  
acids only or whether also chloroanhydrides participate, since  
they also occur on the occasion of the reaction of silicium-  
tetrachloride on the siliciumanhydride which has already  
formed (see formulae!). The acylation of thiophene with the  
above anhydride does not only occur under the presence of

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Tetraacyloxysilanes in Organic Synthesis.

79-12-19/43

X. Comparative Effect of Catalysts on the Occasion of Acylation  
Reaction of Benzene and Thiophene With Tetraacyloxysilanes.

anhydrous zinc beryllium chloride and boron fluoride, with yields of 25,5-46,5 % but also under the presence of tetra- titanium chloride with a yield of 93,5 %. The acylation of benzene with anhydride occurs under the presence of anhydrous aluminium chloride, as well as of anhydrous iron chloride. No acylation of benzene takes place under the presence of anhydrous zinc chloride, beryllium chloride, boron fluoride and titanium tetrachloride. The acylation of benzene and thiophen leads to the same results in the solvent with pure mixed anhydride of silicic and acetic acid, gained from siliciumtetra- chloride and acetic acid anhydride, also from silicium tetra- chloride and acetic acid with the same results, which is a convincing prove that the acylating agent is in fact the anhydride. On this basis the acylation process of the thiophen nucleus could be proved by means of the mentioned anhydrides. There are 2 tables and 33 references, 9 of which are Slavic.

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Tetraacyloxysilanes in Organic Synthesis.  
X. Comparative Effect of Catalysts on the Occasion of Acylation  
Reaction of Benzene and Thiophene with Tetraacyloxysilanes.

79-12-19/43

ASSOCIATION: Moscow State University  
(Moskovskiy gosudarstvennyy universitet).

SUBMITTED: November 22, 1956

AVAILABLE: Library of Congress

1. Tetraacyloxysilanes - Synthesis
2. Benzene - Chemical reactions
3. Thiophene - Chemical reactions
4. Cyclic compounds - Chemical reactions

Card 3/3

AUTHORS: Yur'yev, Yu. K., Dyatlovitskaya, S. V.,  
Bulavin, L. G.

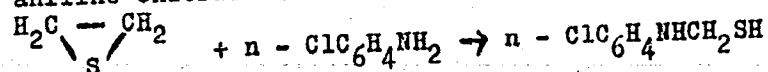
79-12-20/43

TITLE: Ethylene Sulphide in Synthesis of the Heterocyclic Compounds  
with two Hetero-Atoms  
(Etilensul'fid v sinteze geterotsiklicheskikh soyedineniy s  
dvumya geteroatomami).

VI. N - ( $\beta$ -mercaptoethyl) -  $\pi$  - Aniline Chloride and its  
Condensations with Aldehydes, Phosgenes, Carbon Disulphide  
(N - ( $\beta$ -merkaptotetil) -  $\pi$  - khloranilin i kondensatsii yego  
s al'degidami, fosgenom, serouglerodom).

PERIODICAL: Zhurnal Obshchey Khimii 1957, Vol. 27, Nr 12, pp. 3271-3275  
(USSR)

ABSTRACT: In this work the reaction between ethylene sulphide and  
p - aniline halides was investigated. When using  $\pi$  - aniline  
chloride the authors obtained N - ( $\beta$ -mercaptoethyl) -  $\pi$  -  
aniline chloride



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However, it was impossible to carry out the same transposition  
with p - bromine or p - aniline iodide: On the occasion of

Ethylene Sulphide in Synthesis of the Heterocyclic Compounds  
with two Hetero-Atoms.

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VI. N - ( $\beta$ -mercaptoethyl) -  $\pi$  - Aniline Chloride and its  
Condensations with Aldehydes, Phosgenes, Carbon Disulphide.

an attempt to precipitate N - ( $\beta$ -mercaptoethyl) -  $\pi$  - aniline  
bromine by means of distillation an explosion occurred at  
110 - 115° which was the case also with all iodine compounds  
inspite of all possible precautionary measures. This in-  
stability which both compounds must be explained by the mob-  
ility of bromine and the still greater one of iodine which  
gives the possibility that further condensations must occur  
towards the sulphohydro- and aminogroup at increased  
temperature. The spontaneous release of hydrogen halide then  
leads to the explosion. The interaction between the ethylene  
sulphide and p - aniline chloride thus leads to N - ( $\beta$ -  
mercaptoethyl) -  $\pi$  - aniline chloride which on the occasion  
of oxydation with iodine forms a dihydrate  $\beta, \beta'$  - Di - (p-  
chlorophenylamino) diethyldisulphide. N - ( $\beta$ -mercaptoethyl) -  
 $\pi$  - aniline chloride frequently condensates with fat and  
aromatic aldehydes (with formic, propionic, butyric and benzoic  
aldehyde) as well as with phosgenes and with carbonic disulph-  
ide. Thus, 3 -  $\pi$  - chlorophenyl, 2 - methyl - 3 -  $\pi$  - chloro-  
phenyl, 2 - ethyl - 3 -  $\pi$  - chlorophenyl - 2 - propyl -

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Ethylene Sulphide in Synthesis of the Heterocyclic Compounds with two Hetero-Atoms.

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VI. H - ( $\beta$ -mercaptoethyl) .  $\tau$  - Aniline Chloride and its Condensations with Aldehydes, Phosgenes, Carbon Disulphide.

3 -  $\tau$  - chlorophenyl; and 2 - phenyl - 3 -  $\tau$  - chlorophenylthiazolidine as well as also 3 -  $\tau$  - chlorophenylthiazolidine - 2 - and 3 -  $\tau$  - chlorophenylthiazolidinethion - 2 which are not described in technical literature are synthesized.

There are 10 references, 6 of which are Slavic.

ASSOCIATION: Moscow State University  
(Moskovskiy gosudarstvennyy universitet).

SUBMITTED: December 28, 1956

AVAILABLE: Library of Congress

1. Cyclic compounds - Synthesis
2. Cyclic compounds - Condensation reactions

Card 3/3



YU. K. 10. K.

20-2-26/60

**AUTHORS:** Korobitsyna, I. K. , Zhukova, I. G. , Kuvshinova, V. A. ,  
Gaydamovich, N. N. , Yur'yev, Yu. K.

**TITLE:** Synthesis and Isomerization of Enol Acetates of  $\beta$ -Furanidons (Sintez i izomerizatsiya enolatsetatov  $\beta$ -furanidonov)

**PERIODICAL:** Doklady Akademii Nauk SSSR, 1957, Vol. 114, Nr 2, pp. 327-330 (USSR)

**ABSTRACT:** The derivatives of the enolic form of tetrahydrofuranen-3 ( $\beta$ -furanidon) and of its homologues have hardly been investigated at all. The authors of the paper under review, in order to produce the acetylic derivatives of the enolic form, used such ketones of the  $\beta$ -furanidon series in which only one single methylene group stands in the  $\alpha$ -position with respect to the carbonyl group. This made it possible to obtain only one enolic acetate with a position of the double bond that was known in advance. Isopropenylacetate was used as acetylating substance. So far, this type of the interesting  $\beta$ -furanidon derivatives has not been described.

Card 1/3      The authors of the paper under review examined the behavior

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Synthesis and Isomerization of Enol Acetates of  $\beta$ -Furanidons

of these enolic acetates with respect to halogenation and isomerization. At chlorine blowing through 2,2,5,5-tetramethylfuranidon-3-enolacetate, or through its solution in chloroform or absolute ether, there is produced at  $-5^{\circ}$  a mono-chlorine-ketone of the furanidine series, i.e. 4-chlorine-2,2,5,5-tetramethylfuranidon-3. This reaction is of fundamental importance, but it has no preparational significance. One of the most interesting reactions is the isomerization of the thermal or catalytic enolacetate-ketone into  $\beta$ -diketones. If triborofluoride is let through cooled enolic acetate at  $-40$  to  $-20^{\circ}$ , no isomerization takes place. At  $-10$  to  $-5^{\circ}$ , on the other hand, after a certain period of induction a turbulent reaction takes place as well as a total resinification of the reaction mixture. If the same enolic acetate is let through a glass tube, which is filled with wadding of glass and heated up to a temperature of  $500^{\circ}$  (but not below) then anisomerization into 4-acetyl-2,2,5,5-tetramethylfuranidon-3 takes place. At higher temperatures the yield decreases from 36.5 % to 5 - 10 %. As a matter of fact, it is split into a ketone and a ketene. The production of a cupric salt and of the derivatives of the 4-acetyl-2,2,5,5-tetramethyl-

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Synthesis and Isomerization of Enol Acetates of  $\beta$ -Furanidons

furanidon-3 as well as an intense violet coloring by solution of ferric chloride confirm its structure. The spectrum of absorption of this cupric salt is analogous to the spectrum of absorption of the cupric salt of acetylacetone, which is one of the characteristic  $\beta$ -diketones. The experimental part of the paper under review describes in detail the reactions together with yields, constants and methods. There are 6 references, 2 of which are Soviet.

ASSOCIATION: Moscow State University imeni M. V. Lomonosov (Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova)

PRESENTED: January 16, 1957, by B. A. Kazanskiy, Member of the Academy

SUBMITTED: January 12, 1957

AVAILABLE: Library of Congress

Card 3/3

*YUR'YEV Yu. K.*  
KOROBITSYNA, I.K.; ZHUKOVA, I.G.; VORONKOVA, V.V.; YUR'YEV, Yu.K.

Synthesis of 4-oxy-2,2,5,5-tetra-alkyl-3-furanidones by the reduction  
of 4-isonitroso-2,2,5,5-tetra-alkyl-3-furanidones. Dokl. AN SSSR 117  
no.2:237-240 N '57. (MIRA 11:3)

1. Moskovskiy gosudarstvennyy universitet im. M.V. Lomonosova.  
Predstavleno akademikom A.N. Nesmeyanovym.  
(Furan compounds)

AUTHORS: Yur'yev, Yu.K., Rozantsev, E.G., and  
Godovikova, S.N.

SOV/55-58-1-24/33

TITLE: Catalytic Changes of Heterocyclic Combinations. LIV. Change of  
2,3,5 - Trialkyl - Furnadynes Into 2,3,5 - Trialkylthiophanes  
(Kataliticheskiye prevrashcheniya geterotsiklicheskikh soyedineniy.  
LIV. Prevrashcheniye 2,3,5 - trialkilfuranidinov v 2,3,5 - trialkil-  
tiofany)

PERIODICAL: Vestnik Moskovskogo universiteta, Seriya fiziko-matematicheskikh i  
yestestvennykh nauk, 1958, Nr 1, pp 183-186 (USSR)

ABSTRACT: The method of the analytic change of oxygen-containing heterocyclic  
combinations in cycles with other heteroatoms was used success-  
fully for the synthesis of 2,3,5 - trimethyl, 2,5 - dimethyl - 3 -  
ethyl - and 2,5 - dimethyl - 3 - propylthiophane out of  
corresponding trialkylfurnidynes. The obtained 2,3,5 - trialkyl-  
thiophanes are colorless fluids not solvable in water, boiling at  
the normal pressure, and having a characteristic odor.  
There are 12 references, 8 of which are Soviet, 3 American, and  
1 French.

ASSOCIATION: Kafedra organicheskoy khimii (Chair of Organic Chemistry)  
Card 1/2

SOV/63-3-6-32/43

AUTHORS: Yur'yev, Yu.K., Rozantsev, E.G., Gribov, B.G.

TITLE: Synthesis of 2,3,5-Trialkylthiophanes by Catalytic Transformation of 2,3,5-Trialkylfuranidines (Sintez 2,3,5-trialkiltiofanov kataliticheskim prevrashcheniyem 2,3,5-trialkilfuranidinov)

PERIODICAL: Khimicheskaya nauka i promyshlennost', 1958, Vol III, Nr 6, pp 830-831 (USSR)

ABSTRACT: The use of the sulfur-organic compounds from petroleum is an important task for Soviet scientists. The different stages of a synthesis of 2,3,5-trialkylthiophanes from 2,3,5-trialkylfuranidines are shown. Other compounds of this group are presented in a table.

ASSOCIATION: There are 7 Soviet references. Moskovskiy gosudarstvennyy universitet imeni M.V. Lomonosova (Moscow State University Imeni M.V. Lomonosov)

SUBMITTED: April 30, 1958

Card 1/1

5 (3,4)  
AUTHORS:Yur'yev, Yu. K., Rosantsev, E. G.,  
Yegorov, Yu. P.

SOV/55-58-6-27/31

TITLE:

The Infrared Spectra of Thiophene and Its Homologues  
(Infrakrasnyye spektry tiofana i yego gomologov)

PERIODICAL:

Vestnik Moskovskogo universiteta. Seriya matematiki, mekhaniki,  
astronomii, fiziki, khimii, 1958, Nr 6, pp 215 - 222 (USSR)

ABSTRACT:

As the exact determination of sulphur containing petroleum fractions is of a great practical and technological interest V. M. Tatevskiy and one of the authors (Ref 1) tried to analyze the Raman-spectrum of thiophene and of eight of its homologues with the result that they observed in all compounds investigated a characteristic frequency of  $690\text{ cm}^{-1}$  which was ascribed to the fully symmetrical oscillation of the thiophene ring. On the other hand, the spectra of the sulphides with open carbon chains show - in the range between  $600$  and  $700\text{ cm}^{-1}$  - also intensive lines which are ascribed to the valency oscillations of the C-S-bond. (Refs 2-5). Hence, as the Raman spectrum is but roughly indicative, and as it is difficult to decipher it in view of its extensive background, the above authors tried to use the infrared spectrum for identifying the five-member

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## The Infrared Spectra of Thiophene and Its Homologues SOY/55-58-6-27/31

cyclic sulphides. Besides, publications are lacking of data on the infrared spectrum of the thiophene homologues. The task, therefore, consisted in finding out the characteristics of the individual bands of the various connecting groups of the homologues worth an analysis. The infrared spectra were taken of the representatives of the 2-alkyl-thiophene range (alkyl- $C_2H_5$ ,  $C_3H_7$ ,  $C_4H_9$ ) (Fig 1), of the 3-alkyl-thiophenes (alkyl- $C_2H_5$ ,  $C_3H_7$ ,  $C_4H_9$ ,  $C_5H_{11}$ ,  $i-C_5H_{11}$ ,  $C_6H_{13}$ ; Fig 2), the spectrum of the 2,5 dimethyl-thiophene, the representatives of the range of the 2,5 dimethyl-3-alkyl-thiophene (alkyl- $CH_3$ ,  $C_2H_5$ ,  $C_3H_7$ ,  $C_4H_9$ ,  $i-C_4H_9$ ,  $C_5H_{11}$  and  $i-C_5H_{11}$  Fig 3). The first two ranges, but also the last, show in their spectra a repetition of various frequencies which can be employed for characterizing the individual compounds. The valency oscillations of C-S are somewhat lower in the 2-alkyl-thiophenes than in the 3-alkyl-thiophenes ( $715-730$  and  $730-750\text{ cm}^{-1}$ ). With all monoalkylthiophenes the frequency of the annular skeleton was at  $1260\text{ cm}^{-1}$ , whilst with the trialkyl-thiophenes this frequency amounted to  $1250\text{ cm}^{-1}$ . The bands, absent in

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The Infrared Spectra of Thiophene and Its Homologues 307/55-58-6-27/31

the infrared spectrum of the thiophene itself and the bands, all but intensive, in the infrared spectrum of the monoalkyl-thiophenes in the range about  $1370\text{ cm}^{-1}$ , were considerably stronger with the trialkylthiophenes. Furthermore, the intensity of the bands in the ranges  $2930\text{--}2940$  and  $2960\text{ cm}^{-1}$  considerably increased with the increase of the methylene groups and the methyl-groups. Investigations in connection with the infrared spectrum proved that they may be employed advantageously for an analysis of the sulphur-containing petroleum fractions for determining the thiophene and its homologues contained therein. The spectrograms were taken by means of the spectrometer IKS-11. The constant values of the monoalkyl-thiophenes and the 2,5 dimethyl-3-alkyl-thiophenes are compiled in tables 1 and 2. The synthesis of the last mentioned compounds is described briefly. There are 3 figures, 2 tables, and 22 references, 13 of which are Soviet.

ASSOCIATION: Kafedra organicheskoy khimii (Chair for Organic Chemistry)

SUBMITTED: September 19, 1958  
Card 3/3

YUR'YEV, Yu.K.: ROZANTSEV, B.G.; GODOVIKOVA, S.H.

Catalytic conversions of heterocyclic compounds. Part 54: Conversion of 2,3,5-trialkylfuranidines to 2,3,5-trialkylthiophenes. Vest. Mosk.un.Ser.mat.,mekh.,astron.,fiz.,khim. 13 no.1:183-186 '58. (MIRA 11:11)

1. Kafedra organicheskoy khimii Moskovskogo gos. universiteta.  
(Furan) (Thiophene) (Catalysis)

YUR'YEV, YU. K.

79-1-1/63

AUTHORS:

Yur'yev, Yu. K., Belyakova, Z. V.

TITLE:

Tetraacyloxysilanes in Organic Synthesis (Tetraatsiloksi-silany v organicheskom sinteze)  
XIII. Synthesis of N,N-Diethylamid- and N-Arylamid Acids With the Aid of the Silicon Anhydrides of Saturated Heterocyclic Organic Acids (XIII Sintez N,N-dietilamidov i N-arilamidov kislots pomeshch'yu kremneangidridov predel'nykh odnoosnovnykh organicheskikh kislots)

PERIODICAL:

Zhurnal Obshchey Khimii, 1958, Vol. 28, Nr 1, pp. 3-6, (USSR)

ABSTRACT:

In connection with earlier foreign works the authors in the present work for the synthesis of N,N-diethylamides and N-arylamides used tetraacyloxysilanes - mixed anhydrides of orthosilicic acid and organic acids. These latter had been successfully utilized by K. D. Petrov in the acylation of aromatic compounds (of benzene and toluene) instead of anhydrides and chloro-anhydrides of acids. The authors adopted the same course in the acylation of heterocyclic compounds - thiophene, selenophene, pyrrole and indole, as well as in the synthesis of aromatic  $\alpha$ ,  $\beta$  - unsaturated

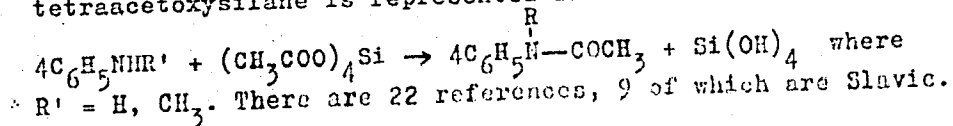
Card 1/3

Tetraacyloxysilanes in Organic Synthesis

79-1-1/63

XIII. Synthesis of N, N-Diethylamid- and N-Arylamid Acids With the Aid of the Silicon Anhydrides of Saturated Monobasic Organic Acids

acids. The reaction of the silicon anhydrides of monobasic organic acids with secondary aliphatic amines may serve as a convenient method of synthesis of N,N-dialkylamid acids. According to this method the authors obtained the N,N-diethylamides of acetic, propionic, n-butyric, n-valerianic, isovalerianic and caproic acids with good yields (60-90%). By the acylation of aniline and methylaniline with the aid of the anhydride of silicic and acetic acid it was shown that the N-arylamides can be synthesized in this manner. In the synthesis of N-substituted acid amides the use of tetraoxysilanes has preference over the chloro-anhydride or the anhydrides of acids. The process of reaction of aniline with tetraacetoxysilane is represented in the following manner:



ASSOCIATION: Moscow State University (Moskovskiy gosudarstvennyy universitet)

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Yur'yev, Yu. K.

79-1-2/63

AUTHORS:

Yur'yev, Yu. K., Zhukova, I. G.

TITLE:

The Catalytic Conversion of Heterocyclic Compounds  
(Kataliticheskiye prevrashcheniya geterotsiklicheskih  
soyedineniy)  
LIII. The Conversion of Oxazole Homologues to Thiazole  
Homologues (LIII. Prevrashcheniya gomologov oksazola v  
gomologi tiazola)

PERIODICAL:

Zhurnal Obshchey Khimii, 1958, Vol. 28, Nr 1, pp. 7-11  
(USSR)

ABSTRACT:

The reaction of oxazoles with hydrogen sulfide which  
should lead to the corresponding thiazoles was not in-  
vestigated. But this reaction, including the derivatives,  
is of high interest for the characteristic of the oxazole  
cycle. The formation of thiazoles from oxazoles in this  
conversion might confirm the chemical proximity of the  
structurally similar cyclic systems to their cyclically  
linked oxygen atom. The investigation performed by the  
authors of the catalytic conversion of the methyl homo-  
logues of oxazole under the influence of hydrogen sulfide

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The Catalytic Conversion of Heterocyclic Compounds  
LIII. The Conversion of Oxazole Homologues to Thiazole Homologues

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showed that the alkyloxazoles, like other five- and six-membered oxygen-containing heterocyclic compounds of an aromatic or aliphatic nature, are subject to a general reaction in which the cyclically bound oxygen is replaced by sulfur. The isomeric dimethyloxazoles (2,4-, 2,5- and 4,5-) and 2,4,5-trimethyloxazole under the influence of hydrogen sulfide upon them in connection with aluminum oxide and at elevated temperatures, like other five- and six-membered oxygen-containing heterocycles, undergo a substitution of the bridge hydrogen by sulfur, where the corresponding dimethylthiazoles (2,5 and 4,5) and 2,4,5-trimethylthiazole are produced. The catalytic conversion of the oxazole-cycle to the thiazole-cycle takes place in the same manner as the analogous conversion of furfuran to thiophene. But it became evident that the oxazole cycle is much more resistant than furfuran with regard to the action of hydrogen sulfide under these conditions. There are 8 references, 2 of which are Slavic.

ASSOCIATION:  
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Moscow State University (Moskovskiy gosudarstvennyy universitet)

AUTHORS: Yur'iyev Yu. Y. Sadovaya, N. K.; 79-28-3-12/61  
Gal'bershtam, M. A.

TITLE: Selenophene Chemistry (Khimiya selenofena)  
X. Synthesis of the 3-methylselenophene-2-Aldehyde and  
2,3-as Well as 2,4-Dimethylselenophene  
(X. Sintez 3-metil'selenofen-2-al'degida i 2,3- i 2,4-  
dimetil'selenofena)

PERIODICAL: Zhurnal Obshchey Khimii, 1958, Vol. 28, Nr 3, pp. 620-624.  
(USSR)

ABSTRACT: In the iodination and reaction with metallo-organic compounds  
of the selenophene nucleus in 3-methylselenophene and 2,4-  
dimethylselenophene (Ref 3) the authors assumed in  
consequence of the analogous structure of seleno- and  
thiophene that there must be also an analogy with respect  
to their chemical properties. Therefore they assumed  
a priori that in the 3-methylselenophene the substitution  
of hydrogen by iodine is accompanied by the formation of  
2-iodo-3-methylselenophene, which after the action of  
lithium phenyl and after carbonization converts to 3-methyl-

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## Selenophene Chemistry

X. Synthesis of the 3-methylselenophene-2-Aldehyde and  
2,3-as Well as 2,4-Dimethylselenophene

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selenophene-2-carboxylic acid. They also concluded that in the iodination of 2,4-dimethylselenophene the substitution of hydrogen by iodine in the position 5 of the nucleus takes place at a formation of 5-iodo-2,4-dimethylselenophene. Therefore, after its metal reaction and carbonization they attributed to the final product the structure 2,4-dimethylselenophene-5-carboxylic acid. The proof had, however, to be given that in the presence of a methyl group in the position 3 of the selenophene nucleus halogenation and mercurization actually take place in the position 2 and that also with 2,4-dimethylselenophene this substitution reaction takes place in position 5. In the present work convincing proof is furnished for this orientation with regard to 3-methyl- and 2,4-dimethylselenophene in the iodination and mercurization. For the synthesis of 2,4-dimethylselenophene the authors used the 2-methylpentene-3, which was added to the reaction with selenium dioxide in the presence of chromium oxide and aluminum oxide, as well as to the reaction with metallic selenium (see mentioned reaction process). From this can be seen that from the step-by-step process only

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Selenophene Chemistry

X. Synthesis of the 3-methylselenophene-2-Aldehyde and  
2,3-as Well as 2,4-Dimethylselenophene

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one 2,4-dimethylselenophene could be obtained. For the synthesis of 2,3-dimethylselenophene the 3-methylselenophene was used the formulation of which leads to an aldehyde to which the structure 3-methylselenophene-2-aldehyde was attributed; this could be supported by experiments. This aldehyde converts to 3-methylselenophene-2-carboxylic acid in oxidation, and to 3-methylselenophene-2-carbinol in reduction. In the investigated reactions of halogenation, mercurization and formulation of 3-methylselenophene it was also proved that the substitution of hydrogen takes place in the position 2 of the selenophene cycle (see formulae).

There are 8 references, 7 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet  
(Moscow State University)

SUBMITTED: February 4, 1957

Card 3/3

AUTHORS:

Yur'yev, Yu. K.; Dyatlovskaya, S. V.

79-28-4-6/60

TITLE:

Ethylene Sulfide in the Synthesis of Heterocyclic Compounds With 2 Hetero Atoms (Etilensul'fid v sinteze geterotsiklicheskikh soedineniy s dvumya geteroatomami) VII. 3-Aryl Thiazolidine-2-Carboxylic Acids and Their Derivatives (VII. 3-ariltiazolidin-2-karbonovyye kisloty i ikh proizvodnyye)

PERIODICAL:

Zhurnal Obshchey Khimii, 1958, Vol. 28, Nr 4, pp. 875-880 (USSR)

ABSTRACT:

The chemistry of thiazolidine and its derivatives was studied specially and in detail with the example of thiazolidine-4-carboxylic acid, which forms an essential part of the structure of the penicillin molecule. However, thiazolidine-2-carboxylic acid itself as well as its derivatives were unknown until very recently. In one of the previous works the authors demonstrated (Ref. 1) that N-( $\beta$ -mercapto ethyl) aryl amines easily condensate with 1,1-dihalogen alkyl and form 3-aryl thiazolidine. In the present work an analogous condensation with dichloro-

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Ethylene Sulfide in the Synthesis of Heterocyclic  
Compounds With 2 Hetero Atoms. VII. 3-Aryl Thiazolidine-  
2-Carboxylic Acids and Their Derivatives

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acetic acid its amide and ether was carried out on the occasion of which the corresponding 3-aryl thiazolidine-2-carboxylic acids, their amides and ethers, and from the latter hydrazides were obtained. Conclusion: 1) Interaction between N-( $\beta$ -mercapto ethyl)-aryl amines and dichloro-acetic acid leads to 3-aryl thiazolidine-2-carboxylic acids not described in technical literature. By this method 3-phenyl-, 3-*m*-tolyl and 3-*m*-anisyl thiazolidine-2-carboxylic acid were obtained. 2) Condensation of N-( $\beta$ -mercapto ethyl)-aryl with dichloro-acetamide which takes place in analogous way leads to amides of the 3-aryl thiazolidine-2-carboxylic acids which are not described in technical literature. By this method amides of the 3-phenyl-, 3-*m*-tolyl-, 3-*o*-tolyl-, 3-*m*-anisyl and 3-*o*-anisyl thiazolidine-2-carboxylic acids were obtained. 3) Condensation of the N-( $\beta$ -mercapto ethyl)-aryl amines with ethyl ether of the dichloro acetic acid leads to ethyl ether 3-phenyl-, 3-*m*-tolyl-, 3-*m*-anisyl- and 3-*o*-anisyl thiazolidine-2-carboxylic acids not described in technical literature and which pass into their hydrazides under the action of the hydrazine hydrate.

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Ethylene Sulfide in the Synthesis of Heterocyclic  
Compounds With 2 Hetero Atoms. VII. 3-Aryl Thiazolidine-  
2-Carboxylic Acids and Their Derivatives

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There are 2 tables and 1 reference, which is Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State  
University)

SUBMITTED: March 14, 1957

Card 3/3

AUTHORS:

Yur'yev, Yu. K.; Makarov, N. B.

79-28-4-8/60

TITLE:

1,4-Oxyketones and 1,4-Diketones in the Catalytic Synthesis of  $\Delta^2$ -Pyrroline-,  $\Delta^2$ -Dihydro-Thiophene Homologs and of the Pyrrole and Thiophene Homologs Respectively (1,4-oksiketony i 1,4-diketony v kataliticheskom sinteze gomologov  $\Delta^2$ -pirrolina,  $\Delta^2$ -digidrotiofena i sootvetstvenno gomologov pirrola i tiofena)

PERIODICAL:

Zhurnal Obshchey Khimii, 1958, Vol. 28, Nr 4, pp. 885-891 (USSR)

ABSTRACT:

As can be seen from previous papers (Refs. 1-8) saturated as well as unsaturated 1,4-diols are introduced in the catalytic synthesis of 5-membered heterocycles. On this occasion the double or triple binding had the central position in the case of the latter, and it was between the second and the third carbon atoms in this system of four carbon atoms. In this connection it was of interest to introduce the most simple 1,4-ketone alcohol as well as 1,4-diketone into the described synthesis. These are bound to react in joint catalytic dehydration with ammonia, with amines or

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1,4-Oxyketones and 1,4-Diketones in the Catalytic  
Synthesis of  $\Delta^2$ -Pyrroline-,  $\Delta^2$ -Dihydro-Thiophene Homologs and of  
the Pyrrole and Thiophene Homologs Respectively

with hydrogen sulfide in the enol form like unsaturated diols with one and correspondingly hydroxyl groups with the carbon atoms of double bond. In the present work the authors used  $\gamma$ -acetopropyl alcohol as representative of the 1,4-oxyketones. It reacted like pentene-2-diol-2,5 in the enol form under the action of ammonia in the presence of aluminum oxide at increased temperature, and produced the 2-methyl- $\Delta^2$ -pyrroline (50%) under the action of aniline-1-phenyl-2-methyl- $\Delta^2$ -pyrroline (58%) and under the action of hydrogen sulfide 2-methyl- $\Delta^2$ -dihydro thiophene (38,5%) (Ref. 9). It was found that in the reactions described the precipitation of water and the closing of the nitrogen or sulfur containing cycles takes place directly by means of joint catalytic dehydration of ammonia, the amines or of hydrogen sulfide as well as of the 1,4-diols and also of the 1,4-oxyketones or 1,4-diketones in their enol form. Consequently the formation of 2-methyl- $\Delta^2$ -dihydro-furan is no obligatory intermediate stage in catalytic synthesis of 2-methyl- $\Delta^2$ -pyrroline from  $\gamma$ -acetopropyl alcohol and ammonia. Conclusion: 1,4-oxyketones and 1,4-diketones can

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1,4-Oxyketones and 1,4-Diketones in the Catalytic  
Synthesis of  $\Delta^2$ -Pyrroline-,  $\Delta^2$ -Dihydro-Thiophene Homologs  
and of the Pyrrole and Thiophene Homologs Respectively

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be used successfully in the catalytic synthesis of 5-membered nitrogen- and sulfur containing heterocyclic compounds. The joint catalytic dehydration of  $\gamma$ -aceto- propyl alcohol with ammonia, aniline, and hydrogen sulfide in the presence of aluminum oxide at 325-330° represents a good method for the corresponding synthesis of 2-methyl-  $\Delta^2$ -pyrroline, 1-phenyl-2-methyl-  $\Delta^2$ -pyrroline, and 2-methyl-  $\Delta^2$ -dihydro thiophene. In the case of joint catalytic dehydration of acetonylacetone with ammonia and hydrogen sulfide at 325° in the presence of aluminum oxide the corresponding 2,5-dimethyl pyrrole, 1-phenyl-2,5-dimethyl pyrrole, and 2,5-dimethyl thiophene form. There are 3 tables and 28 references, 17 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: March 20, 1957

Card 3/3

AUTHORS:

~~Yurlyak, Yu. K.~~ Yelyakov, G. B.,  
Vysokosov, A. N.

79-28-5-41/69

TITLE:

Tetraacyloxy-silanes in Organic Synthesis  
(Tetraatsiloksisilany v organicheskom sinteze).  
XIV. Synthesis of Homologs of Cinnamic Acid (of  
 $\alpha$ -Alkyl- $\beta$ -Phenylacrylic Acids)  
[XIV. Sintez gomologov korichnoy kisloty ( $\alpha$ -alkil- $\beta$ -  
-fenilakrilovykh kislot)]

PERIODICAL:

Zhurnal Obshchey Khimii, 1958, Vol. 28, Nr 5,  
pp. 1302-1306 (USSR)

ABSTRACT:

In the previous paper it was reported (reference 1)  
that tetraacetoxysilane - the mixed anhydride of  
silicic and acetic acid - can be used successfully  
in place of acetic anhydride in the synthesis of  
aromatic  $\alpha$ ,  $\beta$ -unsaturated acids according to Perkin.  
This silane enters condensation with benzaldehyde in  
the presence of usual condensing agents, anhydrous  
sodium acetate as well as potash, in which case cinnamic  
acid is not obtained in smaller yields than in the case

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Tetraacyloxy-silanes in Organic Synthesis.

79-28-5-41/69

XIV. Synthesis of Homologs of Cinnamic Acid (of  $\alpha$ -Alkyl- $\beta$ -Phenylacrylic Acids)

of acetic anhydride. It was therefore of interest to use also the silicic acid anhydrides of other saturated monobasic acids for the synthesis of cinnamic acid homologs. In the present work the silicic acid anhydrides of propionic-, butyric-, valeric-, isovaleric- and capronic acid were introduced, on which occasion a number of  $\alpha$ -alkyl- $\beta$ -phenylacrylic acids were obtained in yields of from 29 ~ 69.5 % (see scheme). Thus the silicic acid anhydrides of the monobasic aliphatic acids can be used in place of the anhydrides of the corresponding acids in the condensation with benzaldehyde, with  $\alpha$ -alkylcinnamic acids resulting from it. When silicic acid anhydrides of propionic- and valeric acid are used, only the corresponding cinnamic acids are obtained; in the case of the silicic acid anhydride of isovaleric acid the  $\beta$ -isopropylstyrene is formed as final product. When using silicic acid anhydrides of butyric acid and capronic acid besides  $\alpha$ -allylcinnamic acids also styrenes are obtained as

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Tetraacyloxy-silanes in Organic Synthesis.

XIV. Synthesis of Homologs of Cinnamic Acid (of  $\alpha$ -Alkyl- $\beta$ -Phenylacrylic Acids) 79-28-5-41/69

side products. There are 14 references, 2 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet  
(Moscow State University)

SUBMITTED: April 3, 1957

Card 3/3

30V/19-28-6-26/63

AUTHORS:

Yur'yev, Yu. K. Yelyakov, G. B., Vysokosov, A. N.

TITLE:

Tetracyloxysilanes in Organic Synthesis (Tetratsiloksel-silany v organicheskom sinteze) XV. The Synthesis of  $\alpha,\beta$ -Unsaturated Acids of the Furfuran- and Thiophene Series (XV. Sintez  $\alpha,\beta$ -nepredel'nykh kislot ryada furana i tiofena)

PERIODICAL:

Zhurnal obshchey khimii, 1958, Vol. 28, Nr 6, pp. 1554-1557 (USSR)

ABSTRACT:

In connection with earlier papers the authors (Ref 1) were interested in bringing about condensation of tetracyloxysilanes with other aldehydes of aromatic type, viz. with furfural and thiophene-2-aldehyde, in order to obtain the  $\beta$ -(furfuryl-2)- and, correspondingly,  $\beta$ -(thienyl-2)-acrylic acid. According to Marckwald (Ref 2) (Markwal'd)  $\beta$ -(furfuryl-2)-acrylic acid (80 %) is obtained according to the usual reaction method recommended by Perkin (Perkin) of furfural and acetic anhydride in the presence of acetic potassium. There are no reports in papers regarding the synthesis of  $\alpha$ -methyl-,  $\alpha$ -propyl- and  $\alpha$ -butyl- $\beta$ -(furfuryl-2)-acrylic acid,

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Tetraacyloxysilanes in Organic Synthesis. XV. The Syn- SOV/9-28-6-26/63  
thesis of  $\alpha,\beta$ -Unsaturated Acids of the Furfuran- and Thiophene Series

according to Ferkin's reaction. In the present paper the following acids were synthesized in the performed condensation of furfurole with silicon anhydrides of the acetic-, propionic-, butyric-, valeric-, isovaleric and capronic acid:  $\beta$ -(furfuryl-2)-acrylic acid and correspondingly  $\alpha$ -methyl-,  $\alpha$ -ethyl-,  $\alpha$ -propyl-,  $\alpha$ -isopropyl and  $\alpha$ -butyl- $\beta$ -(furfuryl-2)-acrylic acid, which proved that the field of application of tetraacyloxysilane in organic synthesis can be extended (see scheme 1). The condensation of thiophene-2-aldehyde with siliconacetic anhydride in the presence of acetic potassium lead to  $\beta$ -(thienyl-2)-acrylic acid. This acid had already been produced by Biderman (Biderman) (Ref 7) with acetic anhydride in the place of the above mentioned anhydride, no yield was mentioned, however. There are 2 tables and 11 references, 1 of which is Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: May 29, 1957

Card 2/3

AUTHORS: Yur'yev, Yu. K., Belyakova, Z. V. SOV/79-28-7-9/64

TITLE: Tetraacyloxy-Silanes in Organic Synthesis (Tetraatsiloksisilany v organicheskom sinteze) XVI. Benzoyloxy-Trichlorosilane and the Reactivity of the Benzoyl Group (XVI. Benzoioksa trikhlorosilan i reaktivnost' benzoiloksal'gruppy)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol. 28, Nr 7, pp. 1755 - 1760 (USSR)

ABSTRACT: In the investigation of the reaction of silicon tetrachloride with benzoic acid the authors found that in the case of an excess of the former (molar ratios 1,7:1 and 2:1) without a solvent, and on a heating within 12 hours with subsequent vacuum distillation the benzoyloxy-trichlorosilane (Formula 1) is obtained almost quantitatively. According to references 2 and 3 organic acids form on the action of excess silicon tetrachloride at 150°-160° in closed vessels the corresponding chlorine anhydrides and the silicagel (II):  $SiCl_4 + 4RCOOH \rightarrow 2RCOCl + [Si(OH)_4] (II)$ . The industrial production of benzoyl

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Tetraacyloxy-Silanes in Organic Synthesis. XVI.

SOV/79-28-7-9/64

Benzoyloxy-Trichlorosilane and the Reactivity of the Benzoyl Group

chloride from benzoic acid and silicon tetrachloride is based on this fact. Contrary to earlier assumptions concerning this reaction process (Schemes III and IV) the data of the present experiments furnish a much more complicated picture. The formation of benzoyl chloride is the result of the conversion of the tetrabenzoyl oxysilane and benzoyloxy-trichlorosilane forming in the reaction mixture, as well as of the subsequent decomposition of the latter at higher temperature. The benzoyloxy group in benzoyloxy-trichlorosilane is more reactive than the chlorine atoms and it is more easily substituted by the ethoxy group on the action of ethylalcohol. There are 1 table and 20 references, 11 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: June 8, 1957

Card 2/3

AUTHORS: Tur'yev, Yu. K., Sedovaya, N. K. SOV/79-28-8-34/66  
 TITLE: Chemistry of Selenophene (Khimiya selenofena) XI,  $\alpha$ -Iodo-selenophenes in the Organo-Iodine Magnesium Synthesis of Selenophene- $\alpha$ -Carboxylic Acids (XI,  $\alpha$ -Yodselenofeny v magniyorganicheskom sinteze selenofen- $\alpha$ -karbonovykh kislot)  
 PERIODICAL: Zhurnal obshchey khimii, 1958, Vol. 28, Nr 8, pp. 2162-2164 (USER)  
 ABSTRACT: Whereas Umezawa (Umezawa) in his paper on the halogenation of the selenophene nucleus (Ref 1), claims that 2-halogen selenophenes, in particular 2-iodoselenophene, show almost no Grignard (Grin'yar) reaction with magnesium, the authors have established, in their present paper, that magnesium can form iodine magnesium selenophenes in dry ether. This reaction, however, requires an activation of the reaction with ethylene bromide and a slow addition of 2-iodoselenophene to magnesium, the ether solution boiling continuously. Carboxylation of the resulting 2-iodomagnesium selenophenes furnishes the corresponding  $\alpha$ -carboxylic acids of the selenophene series, which are identical with those synthesized

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Chemistry of Selenophene. II. *c*-Iodoselenophenes in the Organo-Iodine Magnesium Synthesis of Selenophene-*c*-Carboxylic Acids

SOV/79-28-8-34/66

by the authors on an earlier occasion (Ref 2) (Reaction diagram). The same experiment with 2-bromoselenophene was unsuccessful. The dissociation constants of selenophene-2-carboxylic acid and of benzoic acid were determined in 80 percent ethyl alcohol at 20°, using a potentiometer L.P.-5 (L.P.-5) with a glass electrode. Selenophene-2-carboxylic acid was shown to be ten times as strong as benzoic acid (Table 2). Table 1 illustrates the synthesis of selenophene-*c*-carboxylic acids. There are 2 tables and 3 references, 1 of which is Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: July 5, 1957

Card 2/2



AUTHORS:

Yur'yev, Yu. K., Zaytseva, Ye. L.

SCV/12-28-3-15/66

TITLE:

Chemistry of Selenophene (Khimiya selenofena) XII. 5-Nitroselenophene-2-Aldehyde and 5-Nitroselenophene-2-Carboxylic Acid (XII. 5-Nitroselenofen-2-al'degid i 5-nitro-selenofen-2-karbonovaya kislota)

PERIODICAL:

Zhurnal obshchey khimii, 1958, Vol. 28, Nr 9, p. 2164-2167 (USSR)

ABSTRACT:

One of the authors previously showed that selenophene can be easily formylated by dimethylformamide to form selenophene-2-aldehyde (ref 1). In the present paper, the authors used N-methyl formanilide with good results. These two methods rendered the selenophene-2-aldehyde accessible, and facilitated its nitration, an aim which was attained by the present investigation. The nitration of the aldehyde was effected in acetic anhydride by the action of fuming nitric acid (d 1,5), yielding the dinitrate of 5-nitroselenophene-2-aldehyde. Its yield amounted to 63 % when 5-7 % of concentrated sulfuric acid was added to the nitric acid and to 28,5 % only in all other cases. In the hydrolysis of the

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Chemistry of Selenophene. VII. 5-Nitroselenophene-2- Aldehyde and 5-Nitroselenophene-2-Carboxylic Acid 607/70-28-8-35/66

above diacetate, 5-nitroselenophene-2-aldehyde was obtained, the yield being 68 % (43 % calculated for the selenophene-2-aldehyde introduced in the reaction). By oxidation with potassium dichlorate and sulfuric acid the corresponding carboxylic acid was formed, and by esterification with methyl alcohol its corresponding methyl ester (see reaction diagram). The determination of the dissociation constant of 5-nitroselenophene-2-carboxylic acid showed that it is ten times stronger than p-nitrobenzoic acid, and equals that of o-nitrobenzoic acid. There are 1 table and 7 references, 4 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: July 5, 1957

Card 2/2

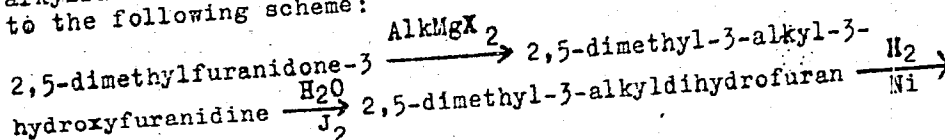
AUTHORS: Yur'yev, Yu. K., Rozantsev, E. G.,  
Godovikova, S. N.

SOV/79-28-8-36/66

TITLE: Synthesis of the 2,5-Dimethyl-3-Alkylfuranidines (Sintez  
2,5-dimetil-3-alkilfuranidinov)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol. 28, Nr 8,  
pp. 2168 - 2171 (USSR)

ABSTRACT: The present paper describes the synthesis of the trialkyl-  
furanidines, which have the alkylradicals in the 2,3 and 5-  
positions. Reports in literature on the 2,5-dimethyl-3-ethyl-  
furanidine only are available (Ref 2). The synthesis is  
carried out by distillation of the 4-ethylhexene-1-ol-5  
with phosphoric acid. The authors synthesized the 2,3,5-tri-  
alkylfuranidines from 2,5-dimethyl-furanidone-3 according  
to the following scheme:



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Synthesis of the 2,5-Dimethyl-3-Alkylfuranidines

SOV/79-28-8-36/66

2,5-dimethyl-3-alkylfuranidine. According to this method 2,3,5-trimethyl-3-hydrofuranidine; 2,5-dimethyl-3-ethyl-3-hydroxyfuranidine and the 2,5-dimethyl-3-propyl-3-hydroxyfuranidine which has not been described, heretofore, were synthesized. The dehydration of the 2,5-dimethyl-3-alkyl-3-hydroxyfuranidine produces a mixture of the dihydrofuran isomers with an admixture of diene hydrocarbons. For the dehydration of the tertiary alcohols of the furanidine series iodine and p-toluene sulfonic acid are the best means. The catalytic hydration of the 2,5-dimethyl-3-alkyldihydrofurans yields 2,5-dimethyl-3-alkylfuranidine. The described synthesis of the 2,3,5-trialkylfuranidines represents a general method of synthesis of the furanidine homologs of this structure. There are 1 table and 7 references, 4 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: July 1, 1957  
Card 2/3

YUR'YEV, Yu.K.; BELYAKOVA, Z.V.; VOLKOV, V.P.

Tetraacyloxy silanes in organic synthesis. Part 17: Acylation of benzene by silicon anhydrides of acetic acid, mono, di and tri-chloroacetic and  $\beta$ -bromopropionic acids. Zhur.ob.khim. 28 no.9: 2372-2376 S '58. (MIRA 11:11)

1. Moskovskiy gosudarstvennyy universitet.  
(Benzene) (Acylation)

YUR'YEV, Yu.K.; DYATLOVITSKAYA, S.V.

Ethylene sulfide in the synthesis of heterocyclic compounds with two heteroatoms. Part 8: Synthesis of 2-(furyl-2')-, 2-(thienyl-2') and 2-(selenyl-2')-3-arylthiazolidines. Zhur.ob.khim. 28 no.9: 2377-2380 S '58. (MIRA 11:11)

1. Moskovskiy gosudarstvennyy universitet.  
(Thiazolidine)

AUTHORS:

Yur'yev, Yu. K., Belyakova, Z. V.,  
Yelyakov, G. B.

SOV/79-28-10-27/60

TITLE:

Tetra-Acyloxy Silanes in Organic Synthesis (Tetraatsiloksi-  
silany v organicheskom sinteze) XVIII. On the Structure  
of the Silicic Anhydrides of the Bivalent Saturated  
Organic Acids (XVIII. O stroenii kremneanglidridov dvukhos-  
novnykh predel'nykh organicheskikh kislots)

PERIODICAL:

Zhurnal obshchey khimii, 1958, Vol 28, Nr 10,  
pp 2766 - 2770 (USSR)

ABSTRACT:

In connection with earlier papers concerning the syn-  
thesis of silicic anhydrides of bivalent organic acids  
from silicon tetrachloride and saturated bivalent acids  
in benzene (Refs 1,2) under the formation of hydrogen  
chloride the problem concerning the structure remained  
unsolved. Based on general considerations the structure  
of a silicic anhydride of the bivalent acid could be  
given by the schemes (I), (II) and (III). With respect  
to the experimental results obtained in the acylation  
of benzene and thiophene with the above mentioned an-  
hydrides it must be mentioned that only ketonic acids and

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Tetra-Acyloxy Silanes in Organic Synthesis. XVIII.

SOV/79-20-10-27/60

On the Structure of the Silicic Anhydrides of the Bivalent Saturated Organic Acids

On account ketones, i.e. the symmetrical dibenzoyl and di(tenoyl-2)-alkanes, were obtained as reaction products, as it is the case in the acylation of the benzene nucleus with these anhydrides. The absence of the diketones in the acylation products of benzene and thiophene with the silicic anhydrides of the bivalent acids was regarded as important, however, not completely sufficient, proof of the structure (III). The further experiment supplied ample proof of the structure (III) since the twice smaller, theoretically necessary amount of hydrogen chloride formed in the reaction of silicon tetrachloride with succinic acid in benzene medium corresponded to the formation of this structure. The data of the analyses of the formed silicon succinic anhydride tend to prove the structure (III). The comparison of the experimental data and the calculated data in the table shows completely that the silicon succinic anhydride synthesized from succinic acid and  $\text{SiCl}_4$  according to the method mentioned corresponds to

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Tetra-Acyloxy Silanes in Organic Synthesis. XVIII. SOV/79-28-10-27/60  
On the Structure of the Silicic Anhydrides of the Bivalent Saturated  
Organic Acids

the structure (III) in which the carboxyl groups are  
maintained in each of the four acid radicals combined with  
the silicon atom. There are 1 table and 10 references,  
3 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State  
University)

SUBMITTED: September 20, 1957

Card 3/3

AUTHORS:

Yur'yev, Yu. K., Vysokosov, A. N.,  
Godovikova, S. N.

SOV/79-28-10-28/60

TITLE:

Tetra-Acyloxy Silanes in Organic Synthesis (Tetraatsiloksi-  
silany v organicheskom sinteze) XIX. Synthesis of the  
3- and 4-Nitro-Cinnamic Acids and Their Homologs of the  
 $\alpha$ -Alkyl- $\beta$ -(Nitrophenyl)-Acrylic Acids (XIX. Sintez 3-1-4-  
nitrokorichnoy kislota i ikh gomologov  $\alpha$ -alkil- $\beta$ -(nitrofenil)-  
akrilovykh kislota)

PERIODICAL:

Zhurnal obshchey khimii, 1958, Vol 28, Nr 10,  
pp 2770 - 2772 (USSR)

ABSTRACT:

In the present paper the condensation of the silicic  
anhydrides of the saturated monovalent organic acids  
with m- and p-nitro-benzaldehyde was carried out.  
The following nitro-cinnamic were obtained in high  
yields: 83,5% 3-nitro-cinnamic-, 89,5% 4-nitro-cinnamic-,  
88% 4-nitro- $\alpha$ -methyl-cinnamic-, 87% 4-nitro- $\alpha$ -ethyl-  
cinnamic-, 47% 4-nitro- $\alpha$ -propyl-cinnamic-, 59% 4-nitro- $\alpha$ -  
isopropyl-cinnamic- and 4-nitro- $\alpha$ -butyl-cinnamic acid.  
Sodium acetate was used as condensing agent in the

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Tetra-Acyloxy Silanes in Organic Synthesis. XIX.  
Synthesis of the 3- and 4-Nitro-Cinnamic Acids and Their Homologs of the  
 $\alpha$ -Alkyl- $\beta$ -(Nitrophenyl)-Acrylic Acids

SOV/79-28-10-28/66

reaction of the m- and p- nitro—benzaldehyde with silicon acetic anhydride; in the condensations with silicic anhydride of the other acids potash (Scheme) was used. The same behaviour of these anhydrides and the anhydrides of organic acids in the Perkins reaction is demonstrated by the fact that their condensation with nitro-benzaldehyde takes place more completely and leads to higher yields of nitro-cinnamic acids than of unsubstituted cinnamic acids. The stabilizing effect of the nitro group of the carbonyl component is shown without any doubt in the intermediate stage of the reaction where the ester of the ortho-silicic acid and of the  $\alpha$ -alkyl- $\beta$ -(nitrophenyl)- $\beta$ -oxyhydro acrylic acid (II) formed from the affiliation product (I) loses the silicic acid more easily under the formation of the compound (III) than it is subjected to the decarboxylation and the separation of silicic acid under the formation of the nitro-styrene homolog (IV). The condensation mentioned above offers high yields of

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Tetra-Acyloxy Silanes in Organic Synthesis. XIX. SOV/79-28-10-28/60  
Synthesis of the 3- and 4-Nitro-Cinnamic Acids and Their Homologs of the  
 $\alpha$ -Aryl- $\beta$ -(Nitrophenyl)-Acrylic Acids

the corresponding nitro-cinnamic acids also in the  
case where the silicic anhydride is formed from an acid  
with a ramified radical. There are 2 tables and 9 refer-  
ences, 2 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State  
University)

SUBMITTED: September 29, 1957

Card 3/3

AUTHORS: Yur'iyev, Yu. K., Sadovaya, N. K.,  
Titov, V. V.

SOV/79-28-11-30/55

TITLE: The Chemistry of Selenophene (Khimiya selenofena)  
XIII. Cyano-Ethylation of the Ketones of the Selenophene  
Series (XIII. Tsianetilirovaniye ketonov ryada selenofena)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol 28, Nr 11,  
pp 3036 - 3041 (USSR)

ABSTRACT: In an earlier paper (Ref 1) the authors succeeded  
to amino-methylate the ketones of the selenophene  
series according to Mannich (Mannikh), which led  
to some  $\beta$ -dimethyl-aminoalkyl-(selenienyl-2)-ketones.  
In the present paper they continue the investigation  
of the reactivity of these ketones and attempted to  
carry out their cyano-ethylation. The cyano-ethylation  
of the 2-acyl selenophenes that do not have alkyl  
radicals in the position 3 takes place quantitatively  
on the action of acrylonitrile in the presence of an  
alkyl alcoholate. In this way the following  $\delta$ -ketone  
nitriles of the selenophene series were obtained:  
1',1',1'-tri(cyano-ethyl)-2-aceto-selenophene,

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The Chemistry of Selenophene. XIII. Cyano-Ethylation  
of the Ketones of the Selenophene Series

SOV/79-28-11-30/55

1',1'-di(cyano-ethyl)-2-propioselenophene, and 1'-methyl-1'-cyano-ethyl-2-propioselenophene. The cyano-ethylation of the 3-methyl-2-acyl selenophene does not take place, apparently because of the difficulties in the spatial arrangement. Only in the case of the 3-methyl-2-acetoselenophene a reaction took place, however, with a smaller yield of 3-methyl-1',1',1'-tri(cyano-ethyl)-2-acetoselenophene. In the alkaline hydrolysis of the  $\delta$ -ketonitriles of the selenophene series  $\delta$ -ketonic acids unknown before in this series were obtained: 1',1',1'-tri(carboxy-ethyl)-2-acetoselenophene, 1',1'-di(carboxy-ethyl)-2-propioselenophene, 1-methyl-1'-carboxy-ethyl-2-propioselenophene and 3-methyl-1',1',1'-tri(carboxy-ethyl)-2-acetoselenophene (Scheme 1). The reduction of the 1'-methyl-1'-cyano-ethyl-2-propioselenophene with formic acid according to the method by A.N.Kost and his collaborators (Ref 8) the 3,3-dimethyl-2-(selenienyl)-piperidon-6 was synthesized (Scheme 2). In the synthesis of the

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The Chemistry of Selenophene. XIII. Cyano-Ethylation  
of the Ketones of the Selenophene Series

SOV/79-28-11-30/55

2-isobutyroselenophene, 3-methyl-2-isobutyroselenophene, and similar selenophenes it became evident that the silicic anhydrides of the saturated ~~mono~~valent organic acids are convenient acylation agents of the selenophene nucleus, which is also the case in the synthesis of the ketones with ramified radicals. There are 12 references, 8 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: September 20, 1957

Card 3/3

AUTHORS: Yur'yev, Yu. K., Mezentsova, N. N. SOV/79-28-11-31/55

TITLE: Chemistry of Selenophene (Khimiya selenofena) XIV.  
Reactions of Selenophene-2-Aldehyde (XIV. Reaktsii selenofen-2-al'degida)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol 28, Nr 11,  
pp 3041 - 3045 (USSR)

ABSTRACT: Continuing earlier papers (Refs 1-3) the authors further investigated the reactions of selenophene-2-aldehyde which are characteristic of aromatic aldehydes. They introduced this aldehyde into the Hantzsch (Ganch) reaction and in a condensation with acetic ester and ammonia obtained the diethyl ester of the 2,6-dimethyl-4-(selenienyl-2')-1,4-dihydropyridine-3,5-dicarboxylic acid (Scheme 1). The condensation of selenophene-2-aldehydes with barbituric acid, malonic acid and malononitrile yielded the selenenal-2-barbituric, selenenal-2-malonic acids, and selenenal-2-malononitrile (Scheme 2). In the cleavage of the 5-(selenenal-2')thiazolidone-4-thione-2 (Ref 2) with

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Chemistry of Selenophene. XIV. Reactions of Selenophene-2-Aldehyde

SOV/79-28-11-31/55

alkali liquor the 2-thione-3-(selenienyl-2')-propionic acid was obtained in good yield, which on the action of hydroxylamine was transformed into the 2-oximino-3-(selenienyl-2')-propionic acid (Scheme 3). The oxime of selenophene-2-aldehyde served as basic product for the selenenyl-2-amine, obtained by the reduction of nitrile. The condensation of this amine with selenophene-2-aldehyde lead to the N-(selenenal-2')-selenenyl-2-amine, with salicylic aldehyde to the N-salicylal-(selenenyl-2')-amine, which could form an inner complex compound  $Cu^{++}$ . There are 2 figures and 3 Soviet references.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: September 30, 1957

Card 2/2

AUTHORS:

~~Yurlov, Ya. Z.~~ Mezentsova, N. N.,  
Vasilevskiy, V. Ya.

SOV/79-28-12-22/41

TITLE:

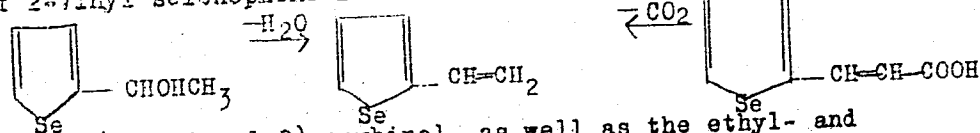
The Chemistry of Selenophene (Khimiya selenofena) XV. 2-Vinyl  
Selenophene (XV. 2-Vinilselenofen)

PERIODICAL:

Zhurnal obshchey khimii, 1958 Vol 28, Nr 12, pp 3262-3265  
(USSR)

ABSTRACT:

Continuing their earlier papers in the field of selenophene chemistry with respect to the selenophene- $\alpha$ -aldehydes (Refs 1-5) the authors synthesized the 2-vinyl selenophene; the catalytic dehydration of methyl-(selenienyl-2)-carbinol turned out to be a better synthesis method than the decarboxylation of  $\beta$ -(selenienyl)-2-acrylic acid, as it led to the synthesis of 2-vinyl selenophene in considerably higher yields:



Methyl-(selenienyl-2)-carbinol, as well as the ethyl- and phenyl-(selenienyl-2)-carbinol were obtained by the reaction of selenophene-2-aldehyde with alkyl and aryl magnesium halides.

Card 1/3

The Chemistry of Selenophene.XV.2-Vinyl Selenophene

SOV/79-28-12-22/41

In the investigation of the effect of various dehydration agents on methyl-(selenienyl-2)-carbinol it was found that in the presence of acid compounds (of potassium bisulfite, p-toluene-sulfonic acid etc.) as well as in the presence of caustic potash the 2-vinyl selenophene formed is almost completely polymerized. In the thermal dehydration the yield of them amounts to 50%, whereas in the dehydration in the vapor phase with aluminum oxide at 200° this figure is 80%. In the above-mentioned decarboxylation reaction the yield amounts to 40% only. 2-vinyl selenophene reacts with diazo methane and forms 4-(selenienyl-2')-pyrazoline, and with dimethyl formamide in the presence of phosphorus oxychloride the  $\beta$ -(selenienyl-2)-acrolein (Scheme 2). In the oxidation of the latter with silver oxide the  $\beta$ -(selenienyl-2)-acrylic acid was obtained, with hydrogen peroxide, however, selenophene-2-carboxylic acid (Scheme 3). There are 1 table and 5 Soviet references.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

Card 2/3

SOV/51-6-1-8/30

**AUTHORS:** Chumayevskiy, N.A., Fatevskiy, V.M. and Yur'yev, Yu.K.

**TITLE:** The Absorption and Raman Spectra of Selenophene and Its Methyl Homologues (Spektry pogloshcheniya i spektry kombinatsionnogo rassseyaniya selenofena i yego metilgomologov)

**PERIODICAL:** Optika i Spektroskopiya, 1969, Vol 6, Nr 1, pp 45-50 (USSR)

**ABSTRACT:** The paper reports studies of the infrared and ultraviolet absorption spectra and Raman spectra of selenophene (I), 2-methylselenophene (II), 3-methylselenophene (III), 2,3-dimethylselenophene (IV), 2,4-dimethylselenophene (V), 3,4-dimethylselenophene (VI). The Raman spectra were obtained on a Steinheil spectrograph (linear dispersion 0.1 mm/Å). The absorption spectra in the infrared were obtained using an IKS-11 spectrometer with LiF (3300-8000 cm<sup>-1</sup>), NaCl (600-3000 cm<sup>-1</sup>) and KBr (400-400 cm<sup>-1</sup>) prisms. An infrared spectrometer VIKSM-3 with a NaCl prism was also used. The ultraviolet absorption spectra were obtained in isooctane using a SF-4 spectrophotometer. Measurements of the infrared spectra, obtained using the VIKSM-3 spectrometer, and of the ultraviolet spectra were carried out at the Optics Laboratory of I.N.S.O.S. of the Academy of Sciences of the U.S.S.R. Selenophene and its homologues studied in the present work

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SOV/51-6-1-8/20

## The Absorption and Raman Spectra of Selenophene and Its Methyl Homologues

had the properties given in a table on p 50. Fig 1 gives the Raman spectra of substances I, III and V (curves a, c and e respectively). Fig 2 gives the infrared absorption spectra of all the six substances studied and Fig 3 gives the corresponding ultraviolet absorption spectra. In all the substances (with the exception of VI) characteristic intense absorption bands appeared in the infrared between 1209 and 1250  $\text{cm}^{-1}$ . The coincidence of certain frequencies and the general similarity of the infrared absorption spectra of thiophene and selenophene and its homologues can be taken as confirmation of the plane structure of selenophene, which belongs to the  $C_{2v}$  type of symmetry. The Raman and the infrared absorption spectra of selenophene agree with the results reported by Gerding et al. (Ref 1). The spectra of selenophene homologues show characteristic frequencies due to vibrations of the substituents (table on pp 46-47). The ultraviolet absorption spectra of selenophene and its homologues obtained in the region 2200-2800 Å did not differ greatly between each other (Fig 3). The ultraviolet spectra of thiophene and its homologues behave in a similar manner. The authors

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SOV/51-6-1-8/30  
The Absorption and Raman Spectra of Selenophene and Its Methyl Homologues

suggest that the infrared absorption and the Raman spectra of selenophene and its homologues may be used for identification of these compounds. There are 3 figures, 2 tables and 11 references, 5 of which are Soviet, 3 French, 2 English and 1 Japanese.

SUBMITTED: March 31, 1958

Card 3/3

S/055/59/000/06/21/027  
B004/B002

AUTHORS: Yur'yev, Yu. K., Rozantsev, E. G.  
TITLE: Dehydration of 2,3,5-Trimethyl-3-oxyfuranidine  
PERIODICAL: Vestnik Moskovskogo universiteta Seriya matematiki, mekhaniki, astronomii, fiziki, khimii, 1959, No. 6, pp. 171-179

TEXT: During the dehydration of 2,3,5-trimethyl-3-oxyfuranidine by means of p-toluene-sulfonic acid, the three isomers shown in the scheme on p 171 may develop, which differ from one another by the position of their double bonds. It was the purpose of this paper to isolate these isomers from the reaction product. The starting substance was produced from 2,5-dimethyl-furanidone-3 according to reference 3. By fractionating the product obtained from the reaction with p-toluene-sulfonic acid, the authors obtained three main fractions (Table 1, Fig. 1). From the first main fraction, 2,3,5-trimethyl- $\Delta^3$ -dihydro-furane was isolated. Its structure was shown by treatment with ozone (development of ketonic acid) and by proving the methylketone group by means of  $KIO_3$ , furthermore by its Raman and infrared spectra (Table 2, Fig. 2), but mainly by means of the infrared spectrum within the range  $2740 - 3100 \text{ cm}^{-1}$  (Fig. 3). If

*Chair of Organic Chem.*

Card 1/3

## Dehydration of 2,3,5-Trimethyl-3-oxofuranidine

S/055/59/000/06/21/027  
B004/B002

clearly shows the absorption bands at  $3065\text{ cm}^{-1}$  of the CH groups of  $\Delta^3$ -dihydro-furane. The ultraviolet spectrum (Fig 4), however, has no absorption maxima, but it shows the absorption which corresponds to substituted ethylene. 2,3,5-trimethyl- $\Delta^2$ -dihydro-furane was isolated from the third main fraction. Its structure was determined by the oxidation with ozone into 2-pentenone-4 and the oxidation of the latter into crotonic acid by means of  $\text{KBrO}$ . Table 2 and figures 2,3,4 also give the corresponding spectra of these compounds. The Raman spectrum shows that the intense line of the double bond is  $1581\text{ cm}^{-1}$  as is the case with the similarly built cyclic vinylether. At  $3095\text{ cm}^{-1}$  the infrared spectrum shows the absorption bands of  $\Delta^2$ -dihydro-furane. The ultraviolet spectrum shows the conjugation of the double bond of the cycle with the free electron pair of oxygen. The third isomer, namely 2,5-dimethyl-3-methylene-furanidine was assumed in the second main fraction. After treatment with 2,4-dinitro-phenyl-hydrazine for the purpose of separating 2,3,5-trimethyl- $\Delta^2$ -dihydro-furane, the substance was again fractionated, and (Table 3) 2,3,5-trimethyl- $\Delta^2$ -dihydro-furane was again obtained. Thus, the dimethyl-methylene compound is unstable and is rearranged into the two trimethyl compounds. There are 4 figures, 3 tables, and 17 references, 5 of which are Soviet.

Card 2/3



7(3)

ADDITIONAL:

Zorulya, A. P., Rozantova, E. B.,  
Peshkova, V. E., Yurlov, M. E.

SOV/75-14-1-3/32

TITLE:

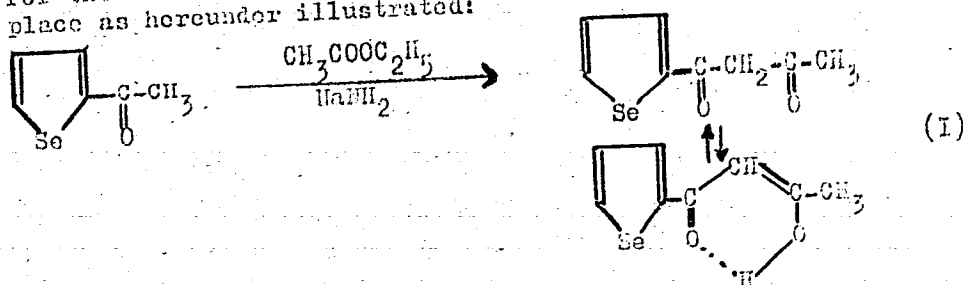
Some Characteristics of Selenenoyl-2-Acetone and Benzoyl  
Acetone (Nekotoryye kharakteristiki selenenoyl-2-atssetona i  
benzoylatssetona)

ABSTRACT:

Zhurnal analiticheskoy khimii, 1959, Vol 14, No 1, pp 17-23  
(USSR)

SYNOPSIS:

The synthesis of selenenoyl-2-acetone (I) which was obtained  
for the first time by the authors of the present paper, took  
place as hereunder illustrated:



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Some Characteristics of  
Selenenoyl-2-Acetone

APPROVED FOR RELEASE: 09/19/2001

CIA-RDP86-00513R001963220014-3

SOV/75-14-1-3/32

The synthesis is very accurately described in the literature. Compound (I) was  
obtained as a viscous straw-yellow oil with boiling  
146 - 146.5°/7 mm, that could be crystallized - melting point  
33 - 33.5° (from alcohol). Benzoyl acetone was synthesized  
according to data found in publications (Ref 4) and had a  
melting point of 58.5 - 59° after repeatedly re-crystallizing  
from ethanol. The absorption spectra of the aqueous solutions  
of both compounds were investigated with different  $\text{pH}$ -values  
between 2 and 11. The respective curves are shown in 2 figures.  
The dissociation constants of both compounds were found in 2  
ways: by computations from the absorption curves and  
graphically from the dependence of the optical density of the  
solutions on the  $\text{pH}$ -value at different wave-lengths. The  
results are specified and show good agreement. A value of  
 $\text{pK}_{\text{diss}} = 8.96 \pm 0.09$  was found for benzoyl acetone, whereas for  
selenenoyl-2-acetone it is  $\text{pK}_{\text{diss}} = 8.55 \pm 0.08$  (ionic strength  
being  $\mu = 0.1$  in the solution). Also the distribution

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SOV/75-14-1-3/32

Some Characteristics of  
Selenenoyl-2-Acetone and Benzoyl Acetone

constants of both compounds in different systems at  $25 \pm 0.1^\circ$   
and  $\mu = 0.1$  were found:

Selenenoyl-2-acetone:  $15 K_{distr} = 2.922 \pm 0.006$  (System  $CHCl_3-H_2O$ )  
 $15 K_{distr} = 3.004 \pm 0.010$  (System  $C_6H_6-H_2O$ )

Benzoyl acetone:

$15 K_{distr} = 3.439 \pm 0.009$  (System  $CHCl_3-H_2O$ )  
 $15 K_{distr} = 3.145 \pm 0.010$  (System  $C_6H_6-H_2O$ )

The distribution constants of both compounds are independent of  
their concentration in the organic phase (benzene, chloroform  
resp.). This is indicative of the fact that neither of the two  
compounds is associated in the aqueous nor in the organic phase.  
There are 5 figures, 6 tables, and 14 references, 3 of which  
are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova  
(Moscow State University imeni M. V. Lomonosov)

SUBMITTED: April 24, 1958

Card 3/3

SOV/79-29-2-16/71

AUTHORS:

Yur'yev, Yu. K., Savosina, M. N.

TITLE:

Alkylation of Benzene With Tetraalkoxy Silanes (Alkilirovaniye benzola tetraalkoksisilanami)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 2, PP 432-435 (USSR)

ABSTRACT:

In the present paper the authors investigate in detail the alkylation of benzene with orthoesters of silicic acid in the presence of aluminum chloride (Ref 6) and state that success depends on the following prerequisites: 1) on the ratio of tetraalkoxy silane to  $AlCl_3$ , 2) on the degree of ramification of the radical in tetraalkoxy silane and 3) on the preparation of tetraalkoxy silane. The data contained in the table show under which conditions ethyl benzene (Experiment 2), sec butyl benzene (Experiments 6 and 7), tert butyl benzene (Experiments 9 and 10) were obtained. The alkylation of benzene was carried out with impure and pure tetraalkoxy silanes, obtained in a benzene medium, in which connection the former gave better yields in alkyl benzenes. The constants obtained agree with those given in publications. With this alkylation no polyalkyl benzenes formed, not even with larger amounts of  $AlCl_3$ , but only mono-

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# Alkylation of Benzene With Tetraalkoxy Silanes

SOV/79-29-2-16/71

alkyl benzenes. On alkylating benzene with tetraalkoxy silanes in the presence of aluminum and iron chloride the same isomerization takes place of the alkyl radical entering the benzene nucleus as is the case of the alkylation of alkyl halides according to G. Gustavson (Ref 7). The results obtained in the work under review made it possible for the authors to suggest the described scheme of benzene alkylation with tetraalkoxy silanes. Thus, monoalkyl benzenes were obtained without impurities of dialkyl benzenes. On alkylating naphthalene under the above conditions, also ethyl benzene (12%) was obtained, besides  $\alpha$ -ethyl naphthalene (24%). There are 1 table and 8 references, 4 of which are Soviet.

ASSOCIATION:

Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED:

December 29, 1957

Card 2/2

SOV/79-29-2-69/71

Yur'yev, Yu. K.

AUTHORS:

Korobitsyna, I. K., Pivnitskiy, K. K.,

TITLE:

Letter to the Editor (Pis'mo v redaktsiyu)  
Synthesis of Mono and Diketones of the Tetrahydropyrane Series From  
Furanidiones-3 and Furanidindiones-3,4 (Sintez mono- i diketonov  
ryada tetragidropirana iz furanidonov-3 i furanidindionov-3,4)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 2, pp 691-693 (USSR)

ABSTRACT:

In continuation of their earlier works and basing on the experience gathered concerning the synthesis and properties of the ketones of the tetrahydrofuran series, the authors carried out the reaction of diazomethane solved in ether (7.2 g, 21 % excess) with 20 g 2,2,5,5-tetramethylfuranidone-3 in ether in the presence of methanol (at 200, 3 days) and obtained 3.95 g 2,2,6,6-tetramethyl tetrahydropyrone-3 (18 % yield, calculated for the ketone introduced into the reaction, besides traces of 2,2,6,6-tetramethyl tetrahydropyrone-4 (Scheme 1) (Ref 2). On the action of diazomethane upon 2,2,5,5-tetraalkyl furanidindione-3,4, the  $\beta$ -diketones forming with the extension of the cycle are methylated by diazomethane, which fact leads to the methyl ethers of 2,2,5,5-tetraalkyl tetrahydropyrandiones-3,5. Thus, for example, the methyl ether of 2,2,6,6-tetramethyl tetrahydropyrandione-3,5

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SOV/79-29-2-69/71

Letter to the Editor. Synthesis of Mono and Diketones of the Tetrahydropyran Series From Furanidones-3 and Furanidindiones-3,4

was obtained on the action of diazomethane solved in ether upon 2,2,5,5-tetramethyl furanidindione-3,4. The hydrolysis of the product with hydrochloric acid yielded 2,2,6,6-tetramethyl tetrahydropyran-3,5, which is soluble in alkali lye. The reaction carried out according to scheme 2 of  $\alpha$ -diketones with diazomethane is described by only one example in publications, namely in the case of camphor quinone (Ref 3).— There are 3 references, 2 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: October 29, 1958

Cará 2/2

CIA-RDP86-00513R001963220014

SOV/79-29-4-8/77

5(3)

AUTHORS:

Yur'yev, Yu. K., Dyatlovitskaya, S. V.

TITLE:

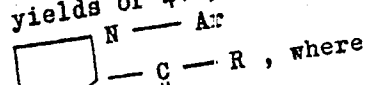
Ethylene Sulfide in the Synthesis of Heterocyclic Compounds With Two Hetero Atoms (Etilensul'fid v sinteze geterotsiklicheskih soyedineniy s dvumya geteroatomami). IX. Synthesis of 2-Acyl-3-aryl Thiazolidines (IX. Sintez 2-atsil-3-ariltiazolidinov)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 4, pp 1083-1086 (USSR)

ABSTRACT:

On the strength of their previous papers (Refs 1-5) the authors could expect that under certain conditions  $\alpha$ -keto aldehydes with N-( $\beta$ -mercapto-ethyl)-aryl amines would primarily react with their aldehyde group, and that in this way the synthesis of thiazolidine ketones hitherto unknown, with one carbonyl group in position 2, would be possible (Ref 6). Methyl glyoxal and phenyl glyoxal were introduced into the condensation with N-( $\beta$ -mercapto-ethyl)-aryl amines, and several 2-acetyl- and 2-benzoyl-thiazolidines were obtained in yields of 41-95%:



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Ethylene Sulfide in the Synthesis of Heterocyclic Compounds With Two Hetero Atoms. IX. Synthesis of 2-Acyl-3-aryl Thiazolidines

$\text{Ar}=\text{C}_6\text{H}_5$ ; o- and p- $\text{CH}_3\text{C}_6\text{H}_4$ ; o- and p- $\text{CH}_3\text{OC}_6\text{H}_4$ ; p- $\text{C}_2\text{H}_5\text{OC}_6\text{H}_4$ ;  $\text{R}=\text{CH}_3$ ,  $\text{C}_6\text{H}_5$ . The absorption spectra in the infrared range of 2-acyl-3-aryl thiazolidines synthesized in this way exhibited a maximum which corresponds to the absorption band of the carbonyl group (Ref 7). The obtained ketones from 2-acyl-3-aryl thiazolidines enter but very difficultly some reactions characteristic of ketones. For instance, they do not react with semicarbazide or thiosemicarbazide, not even on prolonged heating (Ref 8). Oximes of these ketones cannot be obtained owing to a cleavage of the thiazolidine ring observed in the experiments. There are 8 references, 6 of which are Soviet.

ASSOCIATION:

Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED:

February 20, 1958

Card 2/2

SOV/79-29-4-9/77

5(3)

AUTHORS:

Yur'yev, Yu. K., Zaytseva, Ye. L.

TITLE:

Chemistry of Selenophene (Khimiya selenofena). XVI. 4- and 5-Nitroselenophene-2-aldehyde and the Synthesis of Isomeric Mononitroselenophenes (XVI. 4- i 5-Nitroselenofen-2-al'degid i sintez izomernykh mononitroselenofenov)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 4, pp 1087-1093 (USSR)

ABSTRACT:

In connection with the previous paper (1) the authors decarboxylated 5-nitroselenophene-2-carboxylic in the quinoline medium in the presence of pulverized copper, and obtained a yield of 59.5% pure 2-nitroselenophene. Nitroselenophene synthesized in this way melted at 33.5-34° and differed from the preparation obtained by S. Umezawa (Ref 3) by direct nitration of selenophene which melted at 45-46°. As the repeated recrystallization of 2-nitroselenophene synthesized by the authors did not alter its melting point and the elementary analysis pointed to this, they concluded that the preparation of Umezawa was a mixture of 2- and 3-nitroselenophene, and that this result could be supported by investigation of the absorption spectra of isomeric mononitro-

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Chemistry of Selenophene. XVI. 4- and 5-Nitroselenophene-2-aldehyde and the  
Synthesis of Isomeric Mononitroselenophenes

selenophenes obtained in different ways. The product described by Umezawa thus represents, according to the investigations of the authors, a mixture of 2-nitroselenophene (30%) and 3-nitroselenophene (70%). From the nitration of selenophene-2-aldehyde with the nitration mixture a mixture results consisting of 4-nitroselenophene-2-aldehyde, 5-nitroselenophene-2-aldehyde, and 2,4-dinitroselenophene. The first and the latter were separated therefrom. The presence of 5-nitroselenophene-2-aldehyde was confirmed by the absorption spectrum in the ultraviolet range. The oxidation of 4-nitroselenophene-2-aldehyde and the decarboxylation of the resulting 4-nitroselenophene-2-carboxylic acid lead to the formation of 3-nitroselenophene. The absorption spectra in the ultraviolet range of the nitro derivatives of selenophene under investigation are similar to the spectra of the corresponding nitro derivatives of the furan- and thiophene series, which is due to the diene structure of this compound rather than to the nature of the hetero atom. There are 4 figures, 1 table, and 10 references, 2 of which are Soviet.

Card 2/3

5(3)

AUTHORS:

Yur'yev, Yu. K., Belyakova, Z. V.

SOV/79-29-5-11/75

TITLE:

Tetraacyl-oxy-silanes in Organic Synthesis (Tetraatsiloksisilany v organicheskom sinteze).  
20. Synthesis of  $\beta$ -Diketones by Acylation of Malonic and Acetoacetic Ester With Tetraacyl-oxy-silanes (20. Sintez  $\beta$ -diketorov atsilirovaniyem malonovogo i atsetouksusnogo efirov tetraatsiloksisilanami)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 5, PP 1458-1463 (USSR)

ABSTRACT:

An interesting synthesis method of  $\beta$ -diketones of the  $\text{R}(\text{COOCH}_2\text{COR})$  type is the acylation of malonic ester with acid anhydrides in the presence of magnesium oxide or copper acetate catalyst (Ref 16). In the present paper it was investigated whether silicides of monobasic organic acids can be used in this reaction. By their application to the acylation of malonic ester in the presence of magnesium oxide or copper acetate at  $165-170^\circ$  the following  $\beta$ -diketones were obtained in yields of 34-37%: pentanedione-2,4, heptanedione-3,5, nonanedione-4,6, undecanedione-5,7, 2,8-dimethyl nonanedione-4,6 and tridecanedione-6,8. By the action of benzoic acid silicide on malonic ester dibenzoyl methane was obtained in a

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*Moscow State U.*

Tetraacyl-oxy-silanes in Organic Synthesis.

20. Synthesis of  $\beta$ -Diketones by Acylation of Malonic and Acetoacetic Ester with Tetraacyl-oxy-silanes

SOV/79-29-5-11/75

19% yield. Further a method of synthesis for  $\beta$ -diketones of the  $\text{CH}_3\text{COCH}_2\text{COR}$  type was devised. It consists of acylation of the acetoacetic ester with silicides of saturated monobasic organic acids. The reaction takes place under the same conditions and with subsequent ketonic cleavage of the obtained C-acyl derivative of the acetoacetic ester. In this way the following  $\beta$ -diketones were obtained in yields of 34.5-77%: pentanedione-2,4, hexanedione-2,4, heptanedione-2,4, 6-methyl-heptanedione-2,4 and nonanedione-2,4. The yields increase on a change of the ratio acid: acetoacetic ester from 1 : 1 to 2 : 1. In the synthesis of the  $\beta$ -diketones of the

$\text{CH}_3\text{COCH}_2\text{COR}$  type by acylation of the acetoacetic ester with tetraacyl-oxy-silanes it is not always suitable to isolate them in the form of copper salts. On slow distillation sufficiently pure  $\beta$ -diketones are obtained in good yields. The low yields which are obtained on separation on copper salt are probably due to the fact that too little copper salts are formed from the  $\beta$ -diketone mixture with corresponding esters. There are 23 references.

Card 2/3

5(3)

AUTHORS:

Yur'yev, Yu. K., Belyakova, Z. V., Volkov, V. P. SOV/79-29-5-12/75

TITLE:

Tetraacyl-oxy-silanes in Organic Synthesis (Tetraatsiloksisilany v organicheskom sinteze).  
21. Preparation of Acyl-oxy-trichloro Silanes From Acids of Aromatic Nature (21. Polucheniye atsiloksitrikhlorsilancv iz kislot aromaticeskoy prirody)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 5, pp 1463-1470 (USSR)

ABSTRACT:

It was found in the present paper that the homologues of benzoic acid (o- and p-toluic acid),  $\alpha$ -naphthoic acid, substituted benzoic acids (o- and p-chloro benzoic-, o-, m- and p-nitro-benzoic acid) as well as  $\alpha$ -furan carboxylic and  $\alpha$ -thiophene carboxylic acid yield the corresponding acyl-oxy-trichloro silanes under the influence of excess silicon tetrachloride. On longer standing they disproportionate to give tetraacyl-oxy-silanes and silicon tetrachloride. The tendency towards disproportionation rises with an increased strength of the organic acid contained in the acyl-oxy-trichloro silanes (Table 1). On dilution with inert solvents (benzene, ether) acyl-oxy-trichloro silanes can be preserved for some time. Temperature increase accelerates the disproportionation and thus causes a partial decomposition during their distillation in vacuum. The

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